

04/28/99

A

# UTILITY PATENT APPLICATION TRANSMITTAL

## (Large Entity)

*(Only for new nonprovisional applications under 37 CFR 1.53(b))*

04/28/99  
Pocket No.  
U.S. PTO

Total Pages in this Submission

**TO THE ASSISTANT COMMISSIONER FOR PATENTS**

## Box Patent Application

**Washington, D.C. 20231**

Transmitted herewith for filing under 35 U.S.C. 111(a) and 37 C.F.R. 1.53(b) is a new utility patent application for an invention entitled:

**" NOVEL FOAMING COMPOSITIONS AND METHODS FOR MAKING AND USING THE COMPOSITIONS "**

and invented by:

JEFFREY T. PACHL AND DONALD W. TAYLOR AND JAMES W. FREITAG

09/300930  
04/28/99

**If a CONTINUATION APPLICATION, check appropriate box and supply the requisite information:**

☐ Continuation    ☐ Divisional    ☒ Continuation-in-part (CIP) of prior application No.: 09/197,124

Which is a:

☐ Continuation    ☐ Divisional    ☒ Continuation-in-part (CIP) of prior application No.: 09/081,967

Which is a:

☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No.:

Enclosed are:

## Application Elements

1. ☒ Filing fee as calculated and transmitted as described below
2. ☒ Specification having 55 pages and including the following:
- a. ☒ Descriptive Title of the Invention
  - b. ☒ Cross References to Related Applications (*if applicable*)
  - c. ☐ Statement Regarding Federally-sponsored Research/Development (*if applicable*)
  - d. ☐ Reference to Microfiche Appendix (*if applicable*)
  - e. ☒ Background of the Invention
  - f. ☒ Brief Summary of the Invention
  - g. ☒ Brief Description of the Drawings (*if drawings filed*)
  - h. ☒ Detailed Description
  - i. ☒ Claim(s) as Classified Below
  - j. ☒ Abstract of the Disclosure

# UTILITY PATENT APPLICATION TRANSMITTAL (Large Entity)

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Docket No.  
OB003JP-3

Total Pages in this Submission

## Application Elements (Continued)

3. ☒ Drawing(s) (when necessary as prescribed by 35 USC 113)
- a. ☐ Formal Number of Sheets \_\_\_\_\_
- b. ☒ Informal Number of Sheets 1
4. ☒ Oath or Declaration
- a. ☒ Newly executed (original or copy) ☐ Unexecuted
- b. ☐ Copy from a prior application (37 CFR 1.63(d)) (for continuation/divisional application only)
- c. ☐ With Power of Attorney ☐ Without Power of Attorney
- d. ☐ DELETION OF INVENTOR(S)  
Signed statement attached deleting inventor(s) named in the prior application,  
see 37 C.F.R. 1.63(d)(2) and 1.33(b).
5. ☐ Incorporation By Reference (usable if Box 4b is checked)  
The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under Box 4b, is considered as being part of the disclosure of the accompanying application and is hereby incorporated by reference therein.
6. ☐ Computer Program in Microfiche (Appendix)
7. ☐ Nucleotide and/or Amino Acid Sequence Submission (if applicable, all must be included)
- a. ☐ Paper Copy
- b. ☐ Computer Readable Copy (identical to computer copy)
- c. ☐ Statement Verifying Identical Paper and Computer Readable Copy

## Accompanying Application Parts

8. ☒ Assignment Papers (cover sheet & document(s))
9. ☐ 37 CFR 3.73(B) Statement (when there is an assignee)
10. ☐ English Translation Document (if applicable)
11. ☐ Information Disclosure Statement/PTO-1449 ☐ Copies of IDS Citations
12. ☐ Preliminary Amendment
13. ☒ Acknowledgment postcard
14. ☒ Certificate of Mailing

☐ First Class ☒ Express Mail (Specify Label No.): EL056399615US

**UTILITY PATENT APPLICATION TRANSMITTAL**  
**(Large Entity)**

*(Only for new nonprovisional applications under 37 CFR 1.53(b))*

Docket No.  
OB003JP-3

Total Pages in this Submission

**Accompanying Application Parts (Continued)**

15. ☐ Certified Copy of Priority Document(s) *(if foreign priority is claimed)*

16. ☒ Additional Enclosures *(please identify below):*

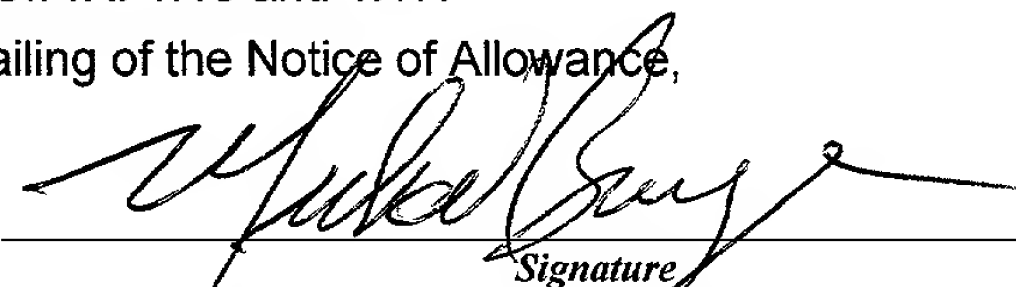
FEE TRANSMITTAL PTO/SB/17

**Fee Calculation and Transmittal**

**CLAIMS AS FILED**

For	#Filed	#Allowed	#Extra	Rate	Fee
Total Claims	20	- 20 =	0	x \$22.00	\$0.00
Indep. Claims	3	- 3 =	0	x \$82.00	\$0.00
Multiple Dependent Claims (check if applicable) <input type="checkbox"/>					\$0.00
BASIC FEE					\$760.00
OTHER FEE (specify purpose) _____					\$0.00
TOTAL FILING FEE					\$760.00

- ☒ A check in the amount of **\$760.00** to cover the filing fee is enclosed.
- ☒ The Commissioner is hereby authorized to charge and credit Deposit Account No. **15-0680** as described below. A duplicate copy of this sheet is enclosed.
- ☐ Charge the amount of \_\_\_\_\_ as filing fee.
- ☒ Credit any overpayment.
- ☒ Charge any additional filing fees required under 37 C.F.R. 1.16 and 1.17.
- ☐ Charge the issue fee set in 37 C.F.R. 1.18 at the mailing of the Notice of Allowance, pursuant to 37 C.F.R. 1.311(b).

  
Signature

**MICHAEL K. BOYER - USPTO REG NO 33,085**  
**ATTORNEY FOR APPLICANTS**  
**ORSCHELN MANAGEMENT CO**  
**2000 US HWY 63 SOUTH**  
**MOBERLY, MISSOURI, 65270**  
tel: 660 269-4536  
fax: 660 269-4530  
email: mboyer@orscheln.com

Dated: **28 APRIL 1999**

CC:

NON-PROVISIONAL PATENT APPLICATION  
NOVEL FOAMING COMPOSITIONS AND METHODS FOR MAKING  
AND USING THE COMPOSITION

CROSS REFERENCE TO RELATED PATENT APPLICATIONS

This is a continuation-in-part of U.S. Patent Application Serial No. 09/197,124, filed on November 20, 1998, that is a continuation-in-part of Serial No. 09/081,967, filed on May 20, 1998 and entitled "Novel Foaming Compositions and Methods For Making and Using the Composition". The disclosure of these prior filed patent applications is hereby incorporated by reference.

The subject matter herein claims benefit under 35 U.S.C. 111(a), 35 U.S.C. 119(e) and 35 U.S.C. 120 of U.S. Provisional Patent Application Serial No. 60/047,273, filed on May 21, 1997, entitled "A Room Temperature Foaming Composition"; and U.S. Provisional Patent Application Serial No. 60/079,205, filed on March 24, 1998, entitled "Novel Foaming Compositions and Methods For Making and Using the Compositions". The disclosure of the aforementioned Provisional Patent Applications is hereby incorporated by reference.

The subject matter of the instant invention is also related to Non-Provisional Patent Application Serial Nos. 09/081,966, filed on May 20, 1998 and Serial No. 09/197,107, filed November 20, 1999, both filed in the name of Jeffrey Pachl et al., and entitled "Curable Sealant Composition". The disclosure of these Non-provisional patent applications is hereby incorporated by reference.

FIELD OF THE INVENTION

The invention relates to foam compositions, precursors thereof and methods for making foam compositions and foam containing articles.

BACKGROUND OF THE INVENTION

Foams are employed in a wide range of commercial applications including applications requiring thermal and sound insulation such as automotive and construction environments, among others. In the automotive industry, foams are typically formed in situ,

and can be used to fill cavities such as pillars and rocker panels, and to dampen sound transmission. In situ foam formation has typically been accomplished by using a polyurethane foam based on isocyanate chemistry. Certain polyurethane foam components and by-products thereof are believed to have an undesirable environmental impact.

- 5 Consequently, there is a need in this art for a low-temperature foam which is cost-effective and substantially free of undesirable materials.

### SUMMARY OF THE INVENTION

10 The invention is capable of solving problems associated with conventional foam formulations by providing foam compositions and precursors thereto which do not require the use of isocyanates. The inventive compositions and precursors can thereof reduce, if not eliminate, the presence of conventional undesirable compounds and by-products thereof while providing benefits associated with conventional foams, e.g, sound/vibration dampening, thermal insulation, structure reinforcement, floatation, energy dissipation, among other  
15 benefits. In addition, the inventive foam has a reduced cured and tack time in comparison to conventional polyurethane foams. These properties in turn improve the efficiency of manufacturing processes that employ foam.

20 One aspect of the invention relates to a method of reacting an epoxy compound and a hydrogen donor or acid compound at ambient conditions to produce a foam. This reaction can produce a relatively large exotherm. The heat released by the exothermic reaction can be sufficient to drive an endothermic blowing agent, thus creating a foam virtually instantaneously. In fact, the exothermic reaction can be sufficiently large to cause a blowing agent entrapped within, for example, thermoplastic powders to expand thereby forming a foam.

25 Another aspect of the invention relates to a method of containing the foam during expansion by expanding the foam within a containment or control means. The control means confines the expanding foam and determines the direction of expansion. While any suitable control means can be employed, a polymeric bag or sack is desirable. If desired, the polymer bag comprises an adhesive material, e.g., the bag adhesive is activated by the exothermic  
30 foam reaction and affixes the resultant foam to a substrate. The polymeric bag can be

fabricated from a virtually unlimited array of materials and configured into any desirable shape, e.g., a honeycomb structure, replicating an automotive cavity, etc.

The inventive foam can be employed in a wide array of end-uses. Examples of such uses include thermal insulation such as appliances, e.g., refrigerators, hot water heaters, etc; aircraft; commercial or residential construction such as spray or rigid insulation for walls, doors, cavity/widow sealant, acoustical control, etc.; packing material, e.g., foam-in-place; marine foams; environmental control, e.g., spill containment; footwear; furniture; toy and consumer goods; protective equipment such as pads, helmets, etc.; fluid filtration; transportation industry uses, e.g., sound dampeners, structural supporting material, etc. for cars, trucks and heavy duty vehicles; vehicle repair; gasketing material; medical uses such as casts, emergency immobilization, etc.; artistic medium such as decorative brick/block, figures, etc.; among others.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a graphical representation of the foam reaction rate and temperature as a function of percent acid.

Figures 2A and 2B are a schematic drawings of assemblies that can be employed for dispensing the inventive foam within a defined cavity or area.

#### DETAILED DESCRIPTION

The invention is based, at least in part, on the surprising discovery that superior foam compositions can be produced from epoxy compounds and acids or hydrogen donor compounds, and in particular, a reaction of the epoxy compounds with the acid source.

Moreover, the inventive compositions can be polyurethane and/or isocyanate free. By “free” it is meant that the inventive compositions before or after foaming contain less than about 10 wt.% polyurethane and/or isocyanurates, isocyanate, and in most cases 0 wt.%. While the presence of such compounds does not adversely affect the reaction described below in greater detail, these materials can be obviated by employing the inventive formulations. The instant invention, therefore, provides a foam which can be used with or instead of urethane/isocyanate based foams and foaming systems.



The inventive foam composition is typically obtained from the reaction of one or more foam precursors. The precursor(s) comprise (i) at least one epoxy compound, and (ii) at least one acid source, i.e., a hydrogen donor or an acid, *e.g.*, phosphoric acid, or a compound such as a photoinitiator which can upon activation provide a hydrogen donor or an acid, and (iii) at least one expansion or blowing agent, among other components. An exothermic reaction between the epoxy and hydrogen donor or acid can activate the expansion or blowing agent thereby producing a foam.

The foam precursor(s) can comprise a single phase system that is activated in response to a source of energy, *e.g.*, heat, UV or electron beam or laser radiation, among other energy sources, or a two component system (an A side precursor and a B side precursor) that are contacted together to produce a foam. When a two component system is employed the epoxy and acid source are provided in separate "side" components.

Alternatively, the foam precursor(s) can comprise a two component system that is activated in response to a source of, *e.g.*, heat, UV or electron beam or laser radiation, among other energy sources. The two component system can include an acid source as well as a photoinitiator.

The first component of the precursor(s), an epoxy compound, comprises about 10 to about 80 wt% of the precursor(s). Examples of suitable epoxy compounds include bis-phenol A epoxy, bis-F epoxy, epoxy-modified elastomers, epoxy-modified polybutene, epoxy-modified polybutadiene, epoxy-modified ethylene-propylene-diene rubber (EPDM), cycloaliphatic epoxy, novolack compounds, and mixtures thereof, among others. When a two component system is employed, the epoxy is located on the A-side, or otherwise prevented from prematurely reacting with the acid or other precursors.

The first component of the precursor can be tailored by adding one or more modifiers. For best results, the modifier is solublized by the epoxy or miscible with the epoxy. Examples of suitable modifiers can comprise at least one member selected from the group consisting of styrene and co-polymers thereof, vinyls and co-polymers thereof, elastomers such as nitrile, ethylene acrylic rubber, mixtures thereof, among others compounds that do not adversely impact the exothermic reaction. Some commercially available materials that can be employed as a modifier comprise Kraton® (Shell Chemical), Vamac® (DuPont), Piccolastic®

(Hercules), Phenoxy® (Paphen), SAA® (styrene-allyl-alcohol copolymer (ARCO), G-Cryl® (Henkel), Rohagum® (Rhomtech), acrylate modified acidic adhesion promoting agent (acid functional oligomer, RadCure®), mixtures thereof, among others. Normally, the epoxy modifier comprises about 2 to about 50 wt.% of the composition prior to foaming.

5 A second component of the precursor(s) is the acid source. When a two component precursor system is employed, the acid source is present in the "B side" of the foam precursors. The hydrogen donor or acid usually comprises about 1% to about 30 wt.% of the precursor, and in particular, about 3% to about 15% of precursor B-side precursors. Examples of suitable acid sources include Lewis acids such as sulfonic acids, phosphoric acid, citric  
10 acid, carboxylic acid, glycolic, tannic, 1,2,4,5-Benzenetetracarboxylic acid, citraconic acid, L-(+)-Citrulline, fumaric, maleic, azelaic, oxalic acids, and mixtures thereof, among others. Particularly desirable results have been achieved by employing at least one of sulfonic, phosphoric acids and other acid functional compounds, e.g., acid functional acrylics. Depending upon the desired reaction rate and resultant foam characteristics, a relatively  
15 concentrated acid can be employed. An example of such a concentrated acid comprises a phosphoric acid that is substantially free of water. By "substantially free" it is meant that the acid contains less than about 10 wt.% and normally less than about 5 wt.% water. Substantially water free acid can be obtained by distilling commercially available acids, e.g., 75% phosphoric acid can be concentrated by distillation. If desired, the acid can comprise an  
20 acid functionally equivalent to the hydrogen donor released by a UV photoinitiator, e.g., replace a portion of the photoinitiator with its corresponding acid.

In one aspect of the invention, an acid substantially free of water is employed to obtain a foam precursor that generates foam having improved structural properties, e.g., foam having a flexural strength 50 to 100% greater than many conventional materials such as wooden  
25 particle board. By employing a substantially water free acid as a foam precursor, the resultant foam has a lower expansion and water absorption, and greater structural strength and adhesion, e.g., to a painted or primed metal surface, wood, Formica®, Masonite®, thermoplastics such as polystyrene, among other surfaces. Substantially water free acids can also permit using a wider range of precursors, e.g., non-polyol carriers.



In another aspect of the invention, the hydrogen donor comprises a photo-initiator that becomes active when exposed to a source of energy. While any photoinitiator capable of becoming a hydrogen donor upon activation can be employed, specific examples of a suitable photo-initiators include a UV catalyst such as UVI 6974 (Union Carbide) that is described in greater detail in the aforementioned copending and commonly assigned U.S. Non-Provisional Patent Application Serial Nos. 09/081,966, filed on May 20, 1998 and Serial No. 09/197,107, filed November 20, 1999, both filed in the name of Jeffrey Pachl et al., and entitled "Curable Sealant Composition". When such an initiator is employed, the foam precursors can be utilized in a single phase system.

For example, such a single phase system can be dispensed, exposed to a UV light source or other suitable source of energy that causes the UV catalyst to generate an acid thereby permitting the epoxy reaction to occur. The heat released by the exothermic epoxy reaction in turn activates an expansion or blowing agent, *e.g.*, a hydrocarbon encapsulated within a thermoplastic, thereby producing a foam. While any suitable single or two phase system can be employed, normally a single phase system produces a foam that is thin relative to a two phase system. Similar to other foam precursors, the radiation activated precursors can be modified for controlling the properties of the precursors or resultant foam, *e.g.*, about 3 to about 20 wt. % polystyrene is added to the epoxy component.

The blowing agent can comprise one or more of the blowing agents recognized in the foam-forming field. Example of suitable blowing agents include water, hydrazide, diphenyloxide-4,4-disulphohydrazide, carbonamide, azocarbonamide, hexamethylene diamine carbamate, sodium bicarbonate, dimethyl ether, methyl chloride, carbon dioxide, fluorocarbons such as difluoroethane, tetrafluoroethane, HFC-4310, azeotropes and isomers thereof, among others; and hydrocarbons such as butane, propane, pentane, isopentane, alcohol, isomers thereof; mixtures thereof, among other known blowing agents. Normally, the expansion or blowing agent comprises about 5 to about 40 wt.% of the foam precursor(s). The blowing agent can be present in either the A or B side of a two component system, although the B-side precursor is preferred.

The foam precursor(s) can also include at least one carrier component, *e.g.*, a polyol, and optional components such as thermoplastics. A carrier usually comprises about 20 to

about 40 wt.% of the precursor, *e.g.*, preferably about 30 wt.%. The carrier typically serves to deliver a component, *e.g.*, an acid, expanding agent, catalyst, mixtures thereof, among others for contact with the epoxy. Examples of suitable carriers comprise at least one member selected from the group of polyols including polyester polyols, polyether, polycarbonate and caprolactone; alcohol, polyvinyl alcohol, synthetic or natural oils such as castor, soy, linseed, glycerin and glycols; water, among other carriers that are preferably miscible with the epoxy and mixtures thereof. When a two component precursor system is employed, these carrier materials are typically added to the acid side or "B side" component of the foam precursors

In addition to the aforementioned epoxy modifiers, the components of the precursor can be tailored by adding one or more modifiers in order to control viscosity, improve stability, physical properties, reaction rates, color, odor, among other characteristics. For best results, the modifier is solublized by the carrier or miscible with a carrier. Examples of suitable modifiers can comprise at least one member selected from the group consisting of natural and synthetic oil such as castor, soy, canola, linseed, polybutene, among other oils. For example, by adding castor oil to the carrier, *e.g.*, Part B-polyester polyol, a relatively hard foam can be obtained. Normally, the carrier modifier is used about 2 wt.% to about 50 wt.% of the composition prior to foaming.

Moreover, the density, moisture and temperature resistance among other physical properties of the final foam product can be modified or tailored by adding a thermoplastic, thermoset, plastic or resinous material to the epoxy-containing precursor. While any suitable modifying material can be employed, examples of such modifying materials include dicyandiamide (Dicy (Amicure CG 1400)), ethylene vinyl acetate, polypropylene, polyethylene, rubber, phenoxy resin, phenolics, powdered wax, solid epoxy such as bis-A epoxy or modified epoxy, novalack compounds, mixtures thereof, among others. For example, depending upon the relative concentration of the components of the precursor, polyvinyl alcohol, hydroscopic polyolefin such as modified polypropylene (as well as other suitable materials) can be employed as modifiers and for absorbing steam or water generated by or during the exothermic reaction. About 1 to about 60 wt.% of modifying material can be added relative to the epoxy, *e.g.*, about 2 wt.% of the precursor(s). The modifying material will normally comprise a powder having a particle size less than about 20 microns and a

melting point from about 200 to about 400 F. The modifying material will become fluid and normally melt when exposed to the exothermic reaction temperature. When a two component precursor system is employed, these materials are normally, but not necessarily, combined with the epoxy or "A side".

5           The foam precursors can also include a thermoplastic component that can function to modify the properties of the resultant foam, reduce material cost, increase precursor shelf life, among other desirable results. The thermoplastic component of the foam precursor(s) can comprise at least one member selected from the group consisting of acrylonitrile, polyethylene, phenolic, wax, EVA, polypropylene, GMA, acid modified polyethylene, 10 polybutadiene, modified polyethylene blend (such as Bynel® supplied by DuPont Company), SIS or SBS or SEBS blocked copolymers (such as Kraton® supplied by Shell Chemical), oligomers, polyolefin, hydroxyl or epoxy functional compounds, among other thermoplastic materials that can be dispersed in a foam precursor and have a melting point less than about the aforementioned exothermic reaction and mixtures thereof. Normally, the thermoplastic component of the precursor will comprise about 1% to about 60 wt.% of the precursor. The 15 thermoplastic component can possess any desirable configuration or particle size. In some cases, the thermoplastic component can form a film or skin upon an exterior surface of the foam thereby improving the resistance of the foam to fluids, *e.g.*, water, gasoline, among other fluids.

20           In one particularly useful aspect of the invention, a liquid or gaseous blowing agent is combined with or encapsulated within a thermoplastic particle or powder, *e.g.*, a hydrocarbon encapsulated within an acrylonitrile shell as in Expancel® that is supplied by Expancel Inc., a division of Akzo Nobel Industries. When a two component precursor system is employed, the shells are normally combined on the B side along with the carrier. These shells can, however, 25 be combined with the A side or in a single phase system so long as the composition of the shells is not substantially affected by the epoxy, *e.g.*, the acrylonitrile or vinylidene chloride shells may be soluble within the epoxy. For example, the shells can be fabricated from polyethylene, nylon, EVA, polypropylene, polyolefin, among other materials not soluble in the epoxy component, and mixtures thereof could be present in the epoxy component of a two 30 phase precursor system. The shells are selected to expand, rupture or retain their physical

configuration depending upon whether or not an open or closed cell foam is desired. Specific examples of suitable encapsulated blowing agents comprise at least one member selected from the group of hydrocarbons such as isobutane and isopentane and fluorocarbons such as 1,1-dichloroethene that are encapsulated within a thermoplastic such as 2-methyl 2-propenoic acid methyl ester polymer with 2-propenenitrile and vinylidene chloride polymer and polyvinylidene fluoride. These materials are supplied commercially by Expancel, Inc., a division of Akzo Nobel as Expancel® 051WU, 051DU, 091DU80, 820WU, 820DU, 642WU, 551WU, 551WU80, 461DU or Micropearl® F30D supplied by Pierce and Stevens. These materials can be supplied in either dry or wet form. When the A and B sides are contacted (or in the case of a single phase system exposed to an energy source), the epoxy reacts with the hydrogen or acid thereby releasing heat and causing the expansion agent within the shells to foam. The foam can be characterized by a composite wherein the epoxy reaction product (including of the aforementioned modifying materials) forms a matrix that embeds the expanded shells. Depending upon the physical characteristics desired in the foam, the shells can be open or closed cells.

The foam characteristics can also be modified by adding one or more filler materials to the precursor(s). Conventionally used filler materials comprise at least one of talc, mica, magnesium silicate, oxidized polyethylene, sodium silicate, alcohols, petroleum jelly, aromatic acid methacrylate-mixed half esters, methacrylated polybutadiene, concrete mix (supplied commercially as Quickrete®), arylalkoxy silane, hollow ceramic spheres, inorganic microspheres, dispersants, conventional blowing/expansion agents, flame retardants such as phosphates, borates and halogenated compounds; plasticizers, diluents, pigments, colorants, metal or ceramic powders, antimicrobial agents such as fungicides, fumed silica, abrasive materials, magnetic materials, anti-static or conductive materials, mixtures thereof, among others. If desired calcium carbonate can be added to the foam precursor for increasing the hardness and density of the resultant foam. When included the filler comprises about 1 to about 60 wt.% of the foam precursors.

The inventive foam can be matrix that embeds or contacts other materials in order to obtain a composite structure. The compositing materials can comprise the aforementioned filler materials, previously formed preform or structures, e.g., honeycomb, fibrous mat,

shaped particulates member, honeycomb structures, syntactic materials such as described in U.S. Patent No. 4,568,603 hereby incorporated by reference; among others. The compositing material can be added to a foam precursor and/or introduced when foaming the precursors. In one aspect, the compositing material comprises styrene pellets, e.g., recycled packaging material, that is ground and added to the previously described carrier. These pellets function to reduce weight and cost of the resultant foam. In another aspect, the compositing material comprises a material for improving the compressive strength of the foam and/or spacers for limiting the degree to which the foam can be compressed, e.g., nylon, polyolefins, polyethylene, among other materials. The compressive strength improving materials can be of any suitable form such as cubes, beads, mixtures thereof, among other shapes.

In one aspect of the invention, one or more foam precursors interact to form an intermediate foam precursor. The intermediate foam precursor can correspond to a Part A and/or Part B. The intermediate foam precursor can be contacted with another precursor or another intermediate foam precursor in order to obtain a foam. A carrier such as a polyol, e.g., a polyester polyol, can interact with at least one member selected from the group of an acid source, e.g., phosphoric acid; a modifier, e.g., styrene; among other precursor components. An epoxy can interact with at least one member selected from the group of an acid source, e.g., phosphoric acid; a modifier, e.g., styrene; among other precursors that are miscible with the epoxy. If desired the aforementioned carrier containing intermediate product is contacted with the aforementioned epoxy containing intermediate product to obtain a foam. The intermediate precursor can be self-supporting. The combined intermediate products can produce a gel-like product that in turn is converted to a foam, e.g., the intermediate product can comprise a gel that can be shaped prior to onset of foam formation.

The precursor(s) and/or intermediate products thereof can be pre-blended and stored in separate containers prior to use. To this end, an A-side or first precursor mixture is typically obtained by combining the epoxy and modifying material, e.g., polyvinyl alcohol and polypropylene, and a B-side or second precursor mixture can be obtained by combining the carrier, e.g., a polyol, hydrogen donor/acid and thermoplastic, e.g., encapsulated blowing agent.



The precursor(s) can be produced using any suitable apparatus that imparts an amount of shear sufficient to obtain a substantially homogenous precursor. Examples of suitable apparatus comprise hand mixing, static tube mixtures, the structures described illustrated by Figures 2A and 2B (described below in greater detail), impingement spraying precursors, extrusion, e.g., a twin screw extruder, among other conventional apparatus. Normally, the samples are mixed for about 1 to about 40 seconds depending upon the composition and mixing environment, e.g., a 1:1 A:B composition can be mixed for about 1 to about 10 seconds in a static tube mixer.

The inventive method involves contacting the epoxy compound and acid or hydrogen donor under conditions effective to provide an exothermic reaction. The heat produced from the reaction can then cause the blowing agent(s) to expand in forming the desired foam. For example, where two precursors, A and B are employed, the two compositions can be combined- to obtain a foam by using conventional foam manufacturing equipment. For example, A-side and B-side can be contacted as two high pressure streams within a mixing chamber of an external mix-head. While heat can be added to the precursors, the reaction between "A" and "B" can occur under ambient conditions, e.g., to control viscosity, adjust reaction rate, etc. The ratio of A-side to B-side normally ranges from about 1:1 to about 10:1 or 1:10.

An example of a combined A and B side precursor composition is set forth in the following Table.

TABLE

<u>Chemical Name</u>	<u>Trade Name</u>	<u>Supplier</u>	<u>Wt. %</u>	<u>Equivalent</u>
Cycloaliphatic Epoxy	Uvacure 1500	UCB Radcure	1 - 80	Sartomer-SARCAT® K126
Polyester Polyol	Tone 0301	Union Carbide	0 - 70	-----
Phosphoric Acid	Phos. Acid	J.T. Baker	1 - 20	commodity
Themoplastic	Expancel	Nobel Industries	1 - 50	Pierce & Stevens-Micropearls®

The pH of the A-side component (containing the epoxy compound(s)) is normally about 6 to at least about 8. The pH of the B side of the foam precursor comprising an acid and a carrier is normally about 0.5 to about 4, e.g., the pH of phosphoric acid when mixed with



polyol. Normally, the pH prior to reaction with A-side precursors is about 1.6. The composition and concentration of the foam precursors can be modified to achieve a predetermined reaction rate *e.g.*, by tailoring the concentration of the acid. The affects of the pH or acid concentration of the B side are better understood by reference to Figure 1 which illustrates the affects upon the composition demonstrated in Example 9.

Referring now to Figure 1, Figure 1 is a graphical representation of % acid in the precursor versus foam reaction time and temperature. Figure 1 illustrates that as the acid concentration increases the reaction temperature increases and the reaction time decreases. Figure 1 also illustrates that the precursor can be selected to a predetermined reaction time/temperature. For example, by selecting a higher reaction temperature (higher acid concentration) a wider range of modifying materials can be employed whereas by selecting a slower reaction time (lower acid concentration) the foam has easier handling characteristics.

The viscosity of a foam precursor can be tailored to enhance the resultant foam characteristics. The viscosity of the "A-side" or epoxy component of the foam precursor is normally controlled, for example, so that a modifying material, *e.g.*, a plastic powder, becomes or remains dispersed within the "A- side" precursor. While any suitable viscosity control agent can be employed desirable results can be achieved by using a solid polymer (in particulate form) to produce a foam precursor gel.

Examples of suitable solid polymers comprise at least one member selected from the group consisting of waxes, polyethylene, EVOH, PVOH, fluoropolymers and dispersions thereof such as polytetrafluoroethylene (supplied as Teflon® by the DuPont Company), among others. The viscosity control agent can range in particle size of about 20 to 50 microns, *e.g.*, less than 325 mesh. An example of a controlled viscosity composition comprises about 5 to about 10 wt.% solid epoxy, about 5 to about 15 wt.%, powdered polyethylene and about 25 to about 30 wt.% blowing agent. In addition to viscosity, the characteristics of the foam can be tailored by varying the temperature, pressure, foam pH, foam density, among other parameters known to those skilled in this art. Also, the "A-side" of the system can be thickened into a gel by the addition of a surfactant such as any commercially available liquid detergent or titanate such as Kenrich KRTTS, *e.g.*, about 0.5 to

about 3 wt.% surfactant. This enables a more complete rheological control, included insuring the homogeneity of the system.

As discussed above, the foam can be produced from a single-phase system, *e.g.*, only an "A-side" mixture. An example of such a system comprises an epoxy, a polyol, thermoplastic spheres, modifying materials, phenoxy, polypropylene, mixtures thereof, among other components. This one component system can be heat activated. In other words the system expands by being exposed to elevated temperature, *e.g.*, about 125C. If desired the single phase foam system can be initiated by employing a photo-initiator instead of, or in conjunction with, an elevated temperature. Examples of such initiators comprise at least one member selected from the group consisting of Union Carbide UVI 6974 among others. Normally, the amount of such an initiator corresponds from 0.5 to about 5 wt% of the foam precursor. More details regarding photoinitiators can be found in "Photopolymerization Behavior of Several Cationic Photoinitiators in Catatonically Cured Resin Systems" by Edward Jurczak; that is hereby incorporated by reference.

Single phase systems are especially useful when applied upon a substrate by being sprayed. For example, the single phase system can be sprayed upon an automotive subassembly for reducing the amount of sound transmission to the interior of the car. In a further example, the single phase system can be sprayed upon a first component, *e.g.*, a plastic fascia, exposed to UV to cause foaming and affixed upon a second component, *e.g.*, metal support member, wherein the foam functions to reduce vibrations between the components.

A composite foam structure can be obtained in accordance with the instant invention. A structural modifier such as fibers, particles, rods, tubes, powders, mixtures thereof, among others, can be incorporated as a component of the foam precursor. The structural modifier can be employed for tailoring the chemical and/or physical properties of the resultant foam. Examples of suitable structural modifiers, normally as chopped fibers, ceramic or glass spheres or powders, can comprise at least one of nylon, carbon, carbonates, polymers such as polyethylene and polypropylene, graphite, Kevlar®, Dyneon, ceramic, fiberglass, mineral fillers, *e.g.*, mica, metals, among other materials. The amount of such structural modifiers normally comprises about 1 to about 60 wt.% of the uncured foam precursor.

Any suitable commercially available foam production equipment can be employed for mixing and dispensing the inventive foam precursors to obtain the inventive foam. Examples of such equipment comprises DoPag (ECONO-MIX) supplied by Kirkco Corporation, Monroe, N.C; as well as equipment supplied commercially by Jesco Products Company, Inc, Sterling Heights, MI. Another example comprises using an Econo-Mix pump in combination with an Albion static mix head. The foam precursors can also be mixed by employing a power mix gun such as supplied by Sealant and Equipment Company, Oak Park, MI. If desired, the inventive foam can be expanded within a cavity, *e.g.*, an automotive A pillar, by employing a dispensing apparatus having a replaceable/disposable static mix head. That is, the static mix head can comprise a replaceable plastic tubing having a center piece with a helix or vortex configuration, that is connected to a pump discharge flange and inserted into the cavity for foaming the precursors.

Another static mix head design has a valve type of arrangement that is illustrated in Figures 2A and 2B. Referring now to Figure 2A, Figure 2A illustrates a one-way valve type of arrangement wherein the foam or precursors thereof are introduced or injected via a one-way valve 1 (commonly known as a zerk) that is positioned within a cap 2. Valve 1 can also include a flap or secondary valve 1A that prevents foam from escaping by reverse flow through valve 1. The cap 2 seals or defines one end of a cavity being filled with foam. The cap 2 can include hooks or locking tabs 3 for securing the position of cap 2, *e.g.*, within the so-called A pillar of an automobile thereby permitting foam to be dispensed within the automotive cavity in a controlled manner. Normally, one end of the valve 1 is connected to a mixing zone 4 such as the aforementioned static mixers having helical vanes 5. After traveling through the valve 1 and static mixer 4, the foam is released into the cavity to be filled with foam. The area and direction into which the foam expands can be control and/or defined by using a containment means such as a polymeric bag 6 (the containment means is described below in greater detail).

Referring now to Figure 2B, this type of arrangement provides a longer mixing time for the foam precursors before the foam is released into the cavity to be filled. The arrangement illustrated in Figure 2B can also be employed as a cap 10 to seal or define one end of the cavity to be sealed. After delivering the foam precursors, a mix head 11 or

previously described valves (4 and 5 of Figure 2A). The foam precursors travel through mix head 11 and are released at the opening defined at 12 as foam. The opening 12 can also be within the aforementioned containment means. The caps 1 and 10 can remain associated with the foam product within the cavity. By using such a replaceable mix head, any problems associated with clogged mix heads are avoided. Two pressure streams can also be employed, to converge in a mix chamber or cavity to be foamed and mix action occurs without use of additional mixing apparatus. In addition to the foregoing, the inventive foam composition and precursors thereof can be injected, extruded, shaped, sprayed, cast, molded, among other conventional processes in order to obtain a desirable foam article. The configuration of the foam article can be virtually any shape including continuous shapes such as films or webs, discrete forms, among other shapes.

While the above description emphasizes particular foam compositions, the inventive compositions (and precursors thereof) can include additives such as dyes, fillers, surfactants, pigments, nucleating agents, among other conventional employed foam additives. If desired a pH indicator can be added to the precursor in order to provide a visual detection means for a reaction product. An example of a suitable pH indicator comprises at least one member selected from the group consisting of methyl red, methyl blue, chlorophenol red, bromothymol blue. That is, as the foam precursor react, e.g, acid-epoxy, the acid is consumed thereby changing the pH and causing the pH indicator to change color.

If desired, the inventive composition can be laminated or joined with other articles, e.g., laminated onto metal foil, Mylar, fiberboard, veneer, Formica® etc. In one aspect of the invention, the inventive foam precursors can be applied between two such laminating materials in order to form components that are useful in fabricating furniture. For example, the inventive foam is expanded between two laminating materials, one of which comprises the upper surface (e.g., a wood veneer) and the second the lower surface of a table top. Any excess foam can be removed by conventional methods such as sawing, scraping, etc. The foam imparts structural integrity to the article while reducing weight and fabrication time.

The inventive composition can also be expanded within a control or containment device or bag having a predetermined shape thereby forming a foamed article that replicates the bag, e.g, refer to U.S. Patent Nos. 4,269,890 (Breitling), 4,232,788 (Roth), 4,390,333

(Dubois); the disclosure of each of which is hereby incorporated by reference. When expanding the foam into a bag, the previously described valves illustrated in Figures 2A and B; those supplied commercially by Inflatable Packaging as part no. IP04, or any other suitable delivery means can be employed at the opening in the bag in order to control introduction of the foam into the bag.

For example, a bag replicating a cavity such as an automotive cavity or any other desirable configuration unrolls or expands into the cavity as foam is introduced into the bag via the valve. If desired, the bag may comprise or be coated with a heat sensitive adhesive wherein the heat generated by the exothermic foam reaction activates the adhesive. The adhesive can permanently affix the foam containing bag at any desirable location. The bag can also include predetermined areas having weakened seams or perforations that are designed to rupture as the foam expands thereby directing the expanding foam. Similarly, the bag composition can be selected such that the bag melts when exposed to the foam. The melting bag can direct the expanding foam, form a coating upon the foam, and function as an adhesive, among other utilities. Further, a plurality of bags can be employed wherein one bag is surrounded by another bag. The inner and/or outer bag can possess the aforementioned predetermined properties. Furthermore, the bag can comprise areas having distinct chemical and/or physical properties, *e.g.*, a bag comprising one sheet of polyethylene heat sealed around its periphery to a sheet comprising polybutadiene. At least a portion of the bag can be fabricated from one or members selected from the group consisting of polyethylene, polyester, vinyl, nylon, Surlyn®, ethylene vinyl acetate, styrene -isoprene- styrene, styrene- butadiene -styrene or other blocked copolymers, polybutadiene, among other plastic materials with melt points corresponding to temperature range of reaction, polyamide, modified EVA's, modified polyethylene, modified polybutadiene, GMA, SBR, among other plastic materials suitable for bag or bladder construction and seaming capability. The bag or containment means can be utilized with a wide range of foam compositions in addition to the previously described epoxy containing foams. Examples of foams that can be expanded into the previously described containment bags or means comprise at least one of epoxy amine, acrylic, and phenolic among others.



The foam precursors can be removed from surfaces, equipment, among other articles by employing non-hazardous cleaning materials. An example of suitable cleaning material comprises water, isopropyl alcohol, 2-butoxyethanol and a chelating agent. The cleaning material can be dispensed as an aerosol by using a propellant such as DME, hydrocarbons and carbon dioxide.

Moreover, the inventive foam can be fabricated to possess a substantially uniform or varying density throughout one or more of its dimensions. The ability to tailor foam density in individual articles as well as throughout an article is a marked improvement in the art. Foams having varying densities can be employed for attenuating or focusing sound, various forms of electromagnetic radiation, radar, etc.

While the above description emphasizes a reaction between an epoxy containing compound and one or more acid or hydrogen donor, the inventive method can be employed by employing other polymer systems such as silicones, urethanes, silanes, hydroxyl modified elastomers, hydroxyl or epoxy functional compounds, among others. That is, a polymer system is contacted with an acid that generates an exothermic reaction which in turn activates an expansion or foaming agent.

The following Examples are provided to illustrate not limit the scope of the invention as defined in the appended claims. Unless indicated otherwise, commercially available apparatus and materials were employed in these Examples.

#### EXAMPLE 1

A foam product was produced by mixing a 2-part system (A-side precursor and B-side precursor) through a conventional foam production apparatus comprising a static mixer that was manufactured by Albion (Model No. 535-1 or equivalent). The constituents of the foam were maintained in two separate supplies of materials, an A-side precursor and B-side precursor.

The A-side precursor comprised a blend of the epoxy and the thermoplastic microspheres including a blowing agent, in ratio of 30 parts to 15 (100 parts total). The B-side precursor comprised a blend of the phosphoric acid and the polyol in a 30 part to 50 part ratio (also 100 parts). The feed ratio of A-side precursor to B-side precursor to the mixer head



was 1:1. The pH of the B-side precursors was about 1.6 prior to reaction with A-side precursor.

A pressurized flow through the mixing chamber produced a polymer which rapidly expands and released an amount of exothermic heat sufficient to produce a foam.

#### EXAMPLE 2

The process of Example 1 was repeated with the exception that the ratio of epoxy to thermoplastic microspheres in A-side precursor was 2:1, and the ratio of phosphoric acid to polyol in B-side precursor was 3:5. The feed ratio of A-side precursor to B-side precursor to the mixer head was 3:1.

#### EXAMPLE 3

The process of Example 1 was repeated with the exception that the A-side precursor and B-side precursor components were mixed together by hand (instead of using the static mixer).

#### EXAMPLE 4

This example demonstrates the formation of a composite foam. The process of Example 1 was repeated with the exception that about 5 wt.% polytetrafluoroethylene powder (TEFLON® supplied by the DuPont Company) was added to the A-side precursor composition. The A-side precursor and B-side precursor were contacted in the manner described in Example 1. A composite foam was recovered wherein the composite foam had greater flexibility or pliability in comparison the foam obtained by the process of Example 1.

#### EXAMPLE 5

A two phase system was used to produce a foam. The A-side precursor was composed of epoxy and microspheres in a 2:1 ratio (67 % epoxy, 33.3% microspheres) by weight. (It is noted that for best results, the mix should be used within in 4-8 hours of mixing since epoxy can dissolve certain spheres). The A-side precursor was hand-stirred to a smooth consistency.

The B-side precursor was composed of Polyol (Tone 0301) and Phosphoric acid (10% by weight). The acid was blended into the polyol. A-side precursor to B-side precursor ratio of 1:1 was contacted in a static tube mixer and produced a foam. The ratio of A to B can be from 1:1 to 4:1 depending on acid concentration.

5

#### EXAMPLE 6

A two phase system was used to produce a foam. The A-side precursor comprised an epoxy (UCB-Radcure UVACURE 1500). The B-side precursor was comprised of a polyol (50 wt.% - Tone 0301), phosphoric acid diluted with water (approximately 50% acid in a commercially available solution) at 20%, and 30% microspheres. The spheres were hand-stirred into the polyol to a smooth consistency. The acid mixture was blended by hand-stirred into the sphere- polyol mix. An A-side precursor to B-side precursor ratio of 1:1 was contacted in a static tube mixer and produced a foam. The ratio of A to B can be from 1:1 to 4:1 depending on acid concentration.

10  
15

#### EXAMPLE 7

A two phase system was used to produce a foam. The A-side precursor comprised an epoxy (UCB-Radcure UVACure1500) While the B-side precursor comprised polyol (Tone 0301), polyvinyl alcohol and water blend (PVOH : H2O 3:1 blend that corresponded to 20% of the polyol) and microspheres 30% by weight of polyol and acid can be 10% of total 'B' mixture. The spheres were hand-stirred into the polyol to a smooth consistency. The PVOH and water are hand-stirred. The PVOH/water solution temperature was 140 °F. The PVOH blend was added to the polyol by hand stirring. The acid was hand-stirred into the sphere-PVOH- polyol mix. The A-side precursor to B-side precursor ratio of 1:1 was contacted in a static tube mixer and produced a foam. The ratio of A to B can be from 1:1 to 4:1 depending upon acid concentration.

20  
25

#### EXAMPLE 8

A two phase system was used to produce a foam. The A-side precursor comprised an epoxy (UCB-Radcure UVACure 1500) and a phenoxy resin (Paphen PKHP-200 that

30

corresponded to 25% of A-side precursors, epoxy is 75% of A-side precursors). The B-side precursor comprised 45 % polyol (Tone 0301), 23.5% polyvinyl alcohol (Airvol 203S) and 23.5% microspheres. Phosphoric acid was 10% by wt. of the B-side precursor. Spheres are hand-stirred into the polyol to a smooth consistency. The PVOH, microspheres, and polyol are blended by hand stirring. The phosphoric acid was hand-stirred into the sphere-PVOH-polyol mix. An A-side precursor to B-side precursor ratio of 1:1 was used contacted in a static tube mixer to produce a foam. The A to B ratio can range from 1:1 to 4:1 depending on acid concentration.

#### EXAMPLE 9A

A two phase system, namely an A-side precursor and a B-side precursor, was used to produce a foam. The A-side precursor comprised an epoxy (UCB-Radcure UVACure 1500) 60 wt%, polypropylene powder (Equistar FP 800-00) 20 wt%, polyvinyl alcohol (Airvol 203S) 20 wt%. The B-side precursor comprised polyol (Tone 0301) 60 wt% and microspheres 30%. Phosphoric acid was 10%. Spheres are hand-stirred into the polyol until a smooth consistency was obtained. The microspheres and polyol are blended by hand stirring. The phosphoric acid was hand-stirred into the microspheres and polyol mix. An A-side precursor to B-side precursor ratio of 1:1 was used and contacted in a static tube mixer to produce a foam. The A to B ratio can, however, range from 1:1 to 4:1 depending on acid concentration.

#### EXAMPLE 9B

This Example employed a two phase system wherein the A-side precursor comprised a gel. A two phase system, namely an A-side precursor and B-side precursor, was used to produce a foam. The A-side precursor comprised an epoxy (UCB-Radcure UVACure 1500) 59 wt%, polypropylene powder (Equistar FP 800-00) 20 wt%, polyvinyl alcohol (Airvol 203S) 20 wt% and surfactant (gelling agent) at 1 wt%. The B-side precursor comprised polyol (Tone 301) 60 wt% and microspheres 30%. Phosphoric acid was 10%. Spheres are hand-stirred into the polyol until a smooth consistency was obtained. The microspheres and polyol are blended by hand stirring. The phosphoric acid was hand-stirred into the

microspheres and polyol mix. An A-side precursor to B-side precursor ratio of 1:1 was used and contacted in a static tube mixer to produce a foam. The A to B ratio can, however, range from 1:1 to 4:1 depending on acid concentration.

#### EXAMPLE 10

A bag or containment device approximately 8x8 inches in size and having a one-way valve located on one end of the bag was constructed from two sheets of high density polyethylene film. The seams of the bag were designed to rupture at specific locations, which directs foam expansion into cavity area adjacent to weak seams. The sheets were joined by heating on a TEW Electric Heating Company Ltd sealing apparatus. The seams were selectively reinforced by double sealing or weakened to provide multiple points for foam direction from the same bag. The foam composition demonstrated by Example 9 was introduced into this bag. As the foam expanded, the foam escaped from the bag through the relatively weak seams.

#### EXAMPLE 11

A bag or bladder composed of each of polyethylene, ethylene vinyl acetate, polybutadiene were fabricated by using the apparatus described in Example 10. The foam of Example 9 was introduced into these bags. The bags, having a melting point less than the exothermic reaction temperature of the foam, failed and released the foam.

#### EXAMPLE 12

A bag or bladder composed of each of modified EVA (Bynel®), modified polyethylene (Primacor® supplied by Dow Chemical Company), modified butadiene, glycidal methacrylate (GMA) were fabricated by using the apparatus of Example 10. The foam of Example 9 was introduced into these bags. The heat released from the exothermic reaction of the foam caused the bags to melt. The melting bag material adhered to the foam thereby modifying the surface of the foam. The melting bag also adhered the foam to any surrounding surfaces or articles.

### EXAMPLE 13

A bag or bladder composed of each of polypropylene, polyethylene, woven nylon mesh, aluminized fiberglass mesh was fabricated by using the apparatus of Example 10. Each of the bags was further processed to possess multiple perforations (25- 100 holes/in.). The foam of Example 9 was introduced into each of these bags. The perforations allowed the foam to escape in controlled quantities while also generally retaining the shape of the bag.

### EXAMPLE 14

Two bags or bladders, namely an inner and outer bag were fabricated by using the apparatus of Example 10. The inner bag comprised modified butadiene and the outer bag comprised high density polyethylene. The inner bag was placed within the outer bag and the outer bag was sealed. The foam of Example 9 was introduced into the inner bag. Inner bag or bladder melted during the foam reaction. The inner bag was of sufficient size to contain the required amount of mixed foam precursors to fill the out bag. Outer bag construction was of material and size to contain reaction within the cavity.

### EXAMPLE 15

The insertion loss or sound dampening characteristics of the foam produced in accordance with Example 9A was tested in accordance with Society of Automotive Engineers (SAE) J 1400. The sample size was 3x3x10 inches and placed within an E-coated metal channel. An increase in insertion loss corresponds to an increase in sound dampening properties that in turn corresponds to less noise within the passenger compartment of an automobile.

FREQ. (Hz)	INSERTION LOSS (dB)
125	12.5
160	10.6
200	11.4
250	12.0
315	24.5

	400	35.4
	500	46.8
	630	38.4
	800	40.1
5	1000	45.7
	1250	45.1
	1600	49.6
	2000	49.2
	2500	50.1
10	3150	50.9
	4000	55.5
	5000	58.7
	6300	59.2
	8000	64.2

These data illustrate the desirable sound absorbing characteristics of the inventive foam compositions.

#### EXAMPLE 16

The viscosity of the Part A foam precursor fabricated in accordance with Example 9A was tested in accordance with conventional methods and apparatus (Brookfield Viscometer, Spindle 27, Thermal-Cell). The viscosity as a function of temperature is listed below.

RPM	Temp 75F	Temp 110F	Temp 150F
0.5	13,000	8,000	3,000
1	10,500	5,500	2,500
2.5	8,160	3,400	1,600
5	6,680	2,300	1,100
10	5,700	1,800	800
20	4,830	1,480	600
50	3,900	1,250	468
100	3,280	1,100	404



The viscosity of the Part B foam precursor fabricated in accordance with Example 9A was tested in accordance with conventional methods and apparatus (Brookfield Viscometer, Spindle 27, Thermal-Cell). The viscosity as a function of temperature is listed below.

	RPM	Temp 75F	Temp 110F	Temp 150F
5	0.5	22,000	13,000	4,000
	1	20,000	10,000	2,500
	2.5	18,600	7,000	1,600
	5	17,800	5,320	1,300
	10	17,300	4,500	1,100
10	20		4,000	975
	50		3,700	880
	100		3,580	860

#### EXAMPLE 17

This Example illustrates foam formation as a result of being activated by exposure to an energy source, e.g, UV light. A radiation curable foam having the following components was prepared:

COMPONENT	TRADE NAME	SUPPLIER	AMOUNT
Cycloaliphatic epoxy	UVACURE 1500	Radcure	50 wt.%
Polyester polyol	Tone 0301	Union Carbide	40
Mechanical blowing agent	Expancel DU551	Expancel Inc.	9
Sulfonium salt	UVI-6974	Union Carbide	1

The above components were combined as follows. The Uvacure and polyol were added together in a mixing vessel and mixed until the solution was clear. The UVI 6974 was added to the mixture, and mixed until substantially completely dispersed (about 2 minutes). The Expancels spheres were added to the mixture and mixed until substantially lump free. For best results, the minimum amount of mixing time, and shear were employed.

The foam precursors were placed onto a conveyor and exposed to a source of UV light. The method for exposing the precursors to UV light is described in the previously

identified U.S. Patent Application 09/197,107, filed November 20, 1999, both filed in the name of Jeffrey Pachl et al., and entitled "Curable Sealant Composition".

This UV activated foam was modified by adding an acrylic monomer or acrylated oligomer. This modified UV activated foam was prepared as described above and comprised:

COMPONENT	TRADE NAME	SUPPLIER	AMOUNT
Acrylated oligomer	IRR 84	UCB RADCURE	93.5 wt.%
Acid functional			
Oligomer	Ebecryl 170	UCB RADCURE	0.9
Photoinitiator	Darocure 1173	Ciba-Giegy	0.9
Blowing Agent	F30D-Micropearls	Pierce & Stevens	4.7

The resultant foam possessed a pressure sensitive adhesive characteristic. The tacky pressure sensitive characteristic was removed by adding an acrylate compound. A tack-free formulation comprised:

COMPONENT	TRADE NAME	SUPPLIER	AMOUNT
Acrylated oligomer	IRR 84	UCB RADCURE	92.6 wt.%
Acid functional			
Oligomer	Ebecure 170	UCB RADCURE	0.9
Photoinitiator	Darocure 1173	Ciba-Giegy	0.9
Blowing Agent	F30D-Micropearls	Pierce & Stevens	4.7
Acrylate	Sartomer 444	Sartomer	0.9

#### EXAMPLES 18-22

The following Table lists the Components, Trade Names and Suppliers for the foam precursors that were employed in Examples 18 through 22. The foam in Examples 18-22 was prepared by contacting the Part A with the Part B listed in the tables below in a 2.75" diameter by 1.92" height ointment can and mixed by hand. Reaction Time and Temperature were determined in accordance with conventional methods. The percent vertical expansion as well as the shrinkage was determined visually. The Shore A test was conducted using a Type A-2 Shore Durometer Hardness test unit that meets ASTM D2240 requirements. The Shore A

test was conducted about 4 hours after foam formation. The instantaneous peak reading was recorded.

Trade Name	Component	Supplier
Expancel 091DU80 (244F)	Blowing Agent (thermal)	Expancel, Inc.
Expancel 051DU (223F)	Blowing Agent (thermal): 2-methyl 2-propenoic acid methyl ester polymer with 2-propenenitrile and isobutane is the blowing agent	Expancel, Inc.
Expancel 054WU (257F)	Blowing Agent (thermal): 2-methyl 2-propenoic acid methyl ester polymer with 2-propenenitrile and isopentane is the blowing agent	Expancel, Inc.
Expancel 461DU (208F)	Blowing Agent (thermal): 2-methyl 2-propenoic acid methyl ester polymer with 1,1-dichloroethene and 2-propenenitrile and isobutane is the blowing agent	Expancel, Inc.
Expancel 551WU (199F)	Blowing Agent (thermal): 2-methyl 2-propenoic acid methyl ester polymer with 1,1-dichloroethene and 2-propenenitrile and isobutane is the blowing agent	Expancel, Inc.
Expancel 551WU80	Blowing Agent (thermal): 2-methyl 2-propenoic acid methyl ester polymer with 1,1-dichloroethene and 2-propenenitrile and isobutane is the blowing agent	Expancel, Inc.
Expancel 642WU (183F)	Blowing Agent (thermal): 2-methyl 2-propenoic acid methyl ester polymer with 1,1-dichloroethene and 2-propenenitrile and isobutane is the blowing agent	Expancel, Inc.
Expancel 820DU (167F)	Blowing Agent (thermal): 2-methyl 2-propenoic acid methyl ester polymer with 1,1-dichloroethene and 2-propenenitrile and isobutane is the blowing agent	Expancel, Inc.
Expancel 820WU (167F)	Blowing Agent (thermal): 2-methyl 2-propenoic acid methyl ester polymer with 1,1-dichloroethene and 2-propenenitrile and isobutane is the blowing agent	Expancel, Inc.
Vertrel XF	Blowing Agent: 2,3-Dihydroperfluoropentane (Pentane, 1,1,1,2,3,4,4,5,5,5-decafluoro: CF <sub>3</sub> CHFCHFCF <sub>2</sub> CF <sub>3</sub> )	DuPont
Micropearls F30D	Blowing Agent (thermal): isobutane encapsulated in polymer vinylidene chloride	HM Royal (Pierce & Stevens)
Ebecryl 170	Adhesion Promoter: Acrylate modified acidic adhesion promoting agent	UCB Radcure
Amicure CG1400	Dicyandiamide	Air Products
Glycolic Acid (70% Tech)	Technical grade (70%)	DuPont
H <sub>3</sub> PO <sub>4</sub> (>95% conc.)	concentrated grade via distillation of the 75% technical grade from Harcros Chemical	DeNOVUS
HQ54	merchant grade (73%)	PCS
Amberphos-54 (AMMGA)	merchant grade (75%)	PCS
H <sub>3</sub> PO <sub>4</sub> (85% Reagent)	reagent grade (85%)	Fischer Scientific
H <sub>3</sub> PO <sub>4</sub> (75% technical)	technical grade (75%)	Harcros Chemicals
H <sub>3</sub> PO <sub>4</sub> (85% technical)	technical grade (85%)	FMC/Harcros Chemicals
BTL 71001	Elastomer: EVA powder	BTL SR Toledo
MU 760-00	Elastomer: EVA powder: MI=23: MP=187F: VA=19: Particle Size=35 mesh	Equistar
Microthene FA 700-00	Elastomer: HDPE powder: MI=10.5: MP=273F: Particle Size=20 microns	Millennium
Microthene FN 514-00	Elastomer: LDPE powder: MI=70: MP=216F: Particle Size=20 microns	Millennium

Trade Name	Component	Supplier
Microthene FP 800-00	Elastomer: Polypropylene powder: MP=325F: Particle Size=20 microns	Equistar
LIR 403	Elastomer: Rubber: (polyisoprene liquid rubber)	Kuraray Co
Kraton D1107	Elastomer: SIS rubber pellets	Shell Chemical Co
Q325	Calcium carbonate	JM Huber Corp
Quikrete	Concrete mix	Quikrete Co.
Dicaperl CS-10-200	Hollow ceramic spheres	Grefco Inc
Qcel 650-D	Inorganic microspheres	PQ Corporation
SynPro Li Stearate	Li stearate	Ferro
A-C 6702	Oxidized polyethylene	Allied Signal
Airvol 203S	Polyvinyl Alcohol (PVOH)	Air Products
G	Sodium silicate	PQ Corporation
BTL 74001	Versatic acid ester/polyvinyl acetate ester	BTL SR Toledo
AZO 77	Zinc oxide	Morton Meyer
Isopropyl Alcohol (70%)	Alcohol	Commercial
Ethanol	Alcohol: Pure Grain Alcohol	Commercial
Ircospense 2174	Dispersant	Lubrizol
Carbopol EZ-1	Emulsion Thickener	BF Goodrich
#1 Castor Oil	Oil	Acme-Hardesty
Lucant HC-2000	Oil: Hydrocarbon based synthetic oil	Mitsui Chemical
Vaseline	Petroleum Jelly	Chesebrough-Ponds
Indopol L100	Polybutene	Amoco
Unifilm 100HSM	Rheology Control Agent	Troy Chemical
Z6040	Silane	Dow Corning
Z6124	Silane: Arylalkoxy silane	Dow Corning
Dish Soap	Soap	commercial
Boraxo	Soap: Sodium tetraborax decahydrate	Dial Corp
Triton X45	Surfactant	Union Carbide
Texaphor Special	Surfactant: Anionic Surfactant	Henkel
KRTTS	Titanate	Kenrich
Santolink XI-100	Allyl glycidyl ether alcohol resin	Solutia/Monsanto
SB 400	Aromatic acid methacrylate-mixed half ester	Sartomer
Pliolite AC	Copolymer: (styrene-acrylate: powder)	Goodyear
Pliolite S-5A	Copolymer: (styrene-butadiene: powder)	Goodyear
CMD 50859	Epoxy:	Shell Chemical Co
CMD 8750	Epoxy:	Shell Chemical Co
PEP 6180	Epoxy: (epoxy toughener: hydrogenated Bis A: )	Pacific Epoxy
PEP 6210 PA	Epoxy: (epoxy toughener: polyether adduct, epoxy functionality: EEW=210: visc=500 cps)	Pacific Epoxy
Erisys GE-60	Epoxy: (sorbitol glycidyl ether – aliphatic polyfunctional epoxy): liquid: EEW=170: visc=13,000 cps	CVC Specialty Chemicals
Epalloy 5000	Epoxy: Bis A: (epoxidized hydrogenated Bis A resin): EEW=220: visc=1900 cps	CVC Specialty Chemicals
DER 317	Epoxy: Bis A: liquid	Dow Chemical
DER 331	Epoxy: Bis A: liquid	Dow Chemical
DER 736	Epoxy: Bis A: liquid	Dow Chemical
Epon 828	Epoxy: Bis A: liquid	Shell
Uvacure 1500	Epoxy: cycloaliphatic	UCB Radcure
Uvacure 1502	Epoxy: cycloaliphatic	UCB Radcure
Uvacure 1533	Epoxy: cycloaliphatic	UCB Radcure
Cryacure UVR 6128	Epoxy: cycloaliphatic	Union Carbide
K126	Epoxy: cycloaliphatic: (cycloaliphatic	Sartomer

Trade Name	Component	Supplier
	diepoxide)	
Eponex 1510	Epoxy: cycloaliphatic: (cycloaliphatic glycidyl ether): (hydrogenated DGEBA)	Shell Chemical Co
Erisys GE-22	Epoxy: cycloaliphatic: (difunctional cycloaliphatic): (cyclohexanedimethanol diglycidyl ether): EEW=155: visc=60 cps	CVC Specialty Chemicals
Uvacure 1534	Epoxy: cycloaliphatic: cycloaliphatic epoxy-polyol blend	UCB Radcure
Erisys GE-35	Epoxy: Glycidyl ether of castor oil	CVC Specialty Chemicals
Epon SU2.5	Epoxy: Novolac	Shell
Epalloy 8240	Epoxy: Novolac: (epoxidized phenol novolac: liquid): EEW=170: visc=6550 cps: fnc=2.35	CVC Specialty Chemicals
Epon 58005	Epoxy: rubber modified: (40% CTBN)	BF Goodrich
Erisys EMRM-22	Epoxy: rubber modified: (CTBN modified epoxy)	CVC Specialty Chemicals
Tone EC	Monomer: Lactone: 2-oxepanone (6-hydroxyhexanoic acid-e-lactone)	Union Carbide
Santicizer 261	Plasticizer: Alkyl Benzyl Phthalate	Solutia
Santicizer 278	Plasticizer: Alkyl Benzyl Phthalate	Solutia
Santicizer 160	Plasticizer: Butyl Benzyl Phthalate	Solutia
Santicizer 97	Plasticizer: Dialkyl Adipate	Solutia
Santicizer 141	Plasticizer: Flame Retardant: 2-ethyl Diphenyl Phosphate	Solutia
CAPA 316	Polyol	Solvay Interlox Ltd
Tone 0201	Polyol: (caprolactone-based polyol: diol): Hydroxyl #=212:	Union Carbide
Tone 0301	Polyol: (caprolactone-based polyol: triol): Hydroxyl #=560: visc=225 @ 55C	Union Carbide
Arcol E-351	Polyol: (polyether polyol: capped diol): Hydroxyl #=40: visc=507Cp	Arco Chemical Co
Arcol DP-1022	Polyol: (polyether polyol: diol): Hydroxyl #=1200: visc=175cP	Arco Chemical Co
PPG-425	Polyol: (polyether polyol: diol): Hydroxyl #=263: visc=71 cps	Arco Chemical Co
Acclaim Polyol 4220	Polyol: (polyether polyol: monol diol):	Arco Chemical Co
Acclaim Polyol 6300	Polyol: (polyether polyol: monol triol): Hydroxyl #=28: visc=1452cP: fnc=2.94: acid value=0.01	Arco Chemical Co
Arcol LG-650	Polyol: (polyether polyol: triol): Hydroxyl #=650: visc=1059cP	Arco Chemical Co
Ebecryl 81	Polyol: Modified polyester polyol	Radcure
K-Flex 188	Polyol: Polyester Polyol:	King Industries
Desmophen L-951	Polyol: Short chained polyol: Hydroxyl #=265	Bayer
Hycar 1300x40	Rubber	BF Goodrich
Hycar 1300X13	Rubber: (CTBN acrylonitrile liquid rubber)	BF Goodrich
Nipol 1312	Rubber: (liquid nitrile rubber)	Zeon Chemical
CN 301	Rubber: (methacrylated polybutadiene)	Sartomer
R45HT	Rubber: (PBD hydroxyl terminated)	Elf Atochem
Actipol E-16	Rubber: Activated polybutene: (Epoxidized polybutene): Liquid	Amoco
Trilene M-101	Rubber: Epoxidized EPDM	Uniroyal
PBD 605	Rubber: Hydroxyl terminated PBD	Elf Atochem
Kraton L-2203	Rubber: Hydroxyl terminated poly (ethylene/butylene) polymer: Diol	Shell Chemical Co
Ricon 100	Rubber: Styrene PBD: Liquid	Ricon Resins
Ricon 184	Rubber: Styrene PBD: Liquid	Ricon Resins
SAT 010	Silyl	Kaneka
SAT 030	Silyl	Kaneka

Trade Name	Component	Supplier
SAT 200	Silyl	Kaneka
Vertrel XF	2,3-Dyhydroperfluoropentane (Pentane, 1,1,1,2,3,4,4,5,5,5-decafluoro: CF <sub>3</sub> CHFCJFCF <sub>2</sub> CF <sub>3</sub> )	DuPont
Micropearls F30D	Thermal Blowing Agent: isobutane encapsulated in polymer vinylidene chloride	HM Royal (Pierce & Stevens)
H <sub>3</sub> PO <sub>4</sub> (>95% conc)	Phosphoric acid: Took Harcros 75% Technical Grade & distilled to >90% acid concentration	DeNOVUS

The following terms and definitions are referenced in Examples 18-22.

Tin Ointment Can: Dimensions 2.75" d x 1.92" h

- 5 Initial Rxn Time: Time that initial expansion is observed (includes mix time, does not include time to pour part A into part B)

Final Rxn Time: Time for reaction to go to completion (includes "Initial Rxn Time")

- 10 Rxn Temp: The peak temperature observed during the reaction

% Ht Expansion:  $\% HE = [(h_f - h_i) / h_i] \times 100$

Density: Weight of 1 in<sup>3</sup> block of expanded material (g/ in<sup>3</sup>)

- 15 H<sub>2</sub>O Absorption 1:  $100 \times (W_3 - W_2) / (W_2 - W_C)$  : W<sub>C</sub>=weight of aluminum coupon only  
W<sub>2</sub>=weight of coupon+material before submerging in water W<sub>3</sub>=weight of coupon+material after submerging in water: Mix material, apply to a 3"x3" aluminum coupon, allow to cool to room temperature, submerge in water for 24 hrs, wipe off excess and immediately calculate water absorption.

- 20 H<sub>2</sub>O Absorption 2: Take a 1.5 inner diameter x h Polyvinyl chloride (PVC) pipe: use Daubert #2-76GSM paper as the release liner inside the PVC pipe (use some means to cap the bottom so that material does not exude out): mix part A & B and pour into the pipe and allow to  
25 expand and cure: cool to room temperature: cut 1.5" lengths so that the sample size is  $\approx 1.5"$ d x 1.5"h: sand the edges: submerge in water for 24 hrs: remove the sample, wipe off excess water and immediately calculate % water absorption.  $\% \text{ Water Absorption} = 100 \times (W_F - W_I) / W_I$

- 30 H<sub>2</sub>O Absorption 3: "Open Chunk": Mix part A & B in a polystyrene plastic cup: allow to expand and cure: cool to room temperature: Cut a "chunk" of foam from the top surface: submerge in water for 24 hrs: remove the sample, wipe off excess water and immediately calculate % water absorption:  $\% \text{ Water Absorption} = 100 \times (W_F - W_I) / W_I$

- 35 Hardness: Shore A: The foam surface may be irregular: Take highest instantaneous reading from top surface after conditioning at room temperature for 4 hrs minimum



Shrinkage: Rating: 0=none 1=<1mm from edge 2=1-2mm from edge 3=2-3mm from edge 4=3-4mm from edge 5=4-5mm from edge 6=5-6mm from edge (shrinkage is usually not symmetrical: take the largest gap and divide by 2 if it did not shrink equally from the outer perimeter. Other values listed will be visually results: Rating 1=very slight, Rating 2=noticeable, Rating 3 & 4=significant, Rating 5 & 6=very significant

#### EXAMPLE 18

Components Wt. %	SAMPLE NO 1	2	3	4	5
<b>Part A</b>					
Uvacure 1500	30	30	30	30	30
Microthene FP800-00	10	10	10	10	
Airvol 203 S (PVOH)	10	10	10	10	10
Dicaperl CS- 10-200					10
<b>Part B</b>					
Tone 0301 (Polyol)	29.65	29.65	29.65	29.65	29.65
Micropearls F30D	14.85	10	12	16	14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5	5.5	5.5	5.5	5.5
Rxn Time	59 sec	57 sec	56	57 sec	53 sec
Rxn Temp	288 F	291 F	289 F	265 F	292 F
% Vertical Expansion	487%	413%	434%	468%	482%
Hardness - Shore A	20	19	20	21	17
Shrinkage	None	None	None	None	Very Slight

Components Wt. %	SAMPLE NO 6	7	8	9
<b>Part A</b>				
Uvacure 1500	30	20	30	30
Microthene FP800-00	10	10	10	
Airvol 203 S (PVOH)	10	10	10	10
Hycar 1300x13	10			
PEP 6180		10	5	
<b>Part B</b>				
Tone 0301 (Polyol)	29.65	29.65	29.65	29.65
Micropearls F30D	14.85	14.85	14.85	14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5	5.5	5.5	5.5

Rxn Time	65 sec	64 sec	58 sec	61 sec
Rxn Temp	265 F	238 F	289 F	270 F
% Vertical Expansion	385%	205%	404%	528%
Hardness – Shore A	22	23	26	18
Shrinkage	Noticeable	Noticeable	Very Slight	Very Slight

Components Wt. %	SAMPLE NO 10	11	12	13	14
<b>Part A</b>					
Uvacure 1500	30	30	20	30	30
Microthene FP800-00			10	10	
Airvol 203 S (PVOH)	20	10	10	10	10
Microthene FN514-00		10			10
DER 317			10		
Microthene FA700-00					10
<b>Part B</b>					
Tone 0301 (Polyol)	29.65	29.65	29.65	20	29.65
Micropearls F30D	14.85	14.85	14.85	14.85	14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5	5.5	5.5	5.5	5.5
CN 301				9.65	
Rxn Time	55 sec	50 sec	56	40 sec	51 sec
Rxn Temp	276 F	274 F	242 F	268 F	277 F
% Vertical Expansion	494%	396%	226%	361%	388%
Hardness – Shore A	16	23	22	42	22
Shrinkage	None	Very Slight	VerySlight	Very Slight	Very Slight

Components Wt. %	SAMPLE NO 15	16	17	18
<b>Part A</b>				
Uvacure 1500	30	30	30	15
Microthene FP800-00				10
Airvol 203 S (PVOH)	10	10	10	10
Equistar MU 76000	10			
AC 6702		10		
BTL 71001			10	
Cryacure UVR 6128				15
<b>Part B</b>				

Tone 0301 (Polyol)	29.65	29.65	29.65	29.65
Micropearls F30D	14.85	14.85	14.85	14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5	5.5	5.5	5.5
CN 301				
Rxn Time	55 sec	50 sec	49 sec	56 sec
Rxn Temp	271 F	280 F	292 F	259 F
% Vertical Expansion	406%	415%	519%	326%
Hardness – Shore A	19		19	15
Shrinkage	Very Slight	Very Slight	None	Very Slight

Components Wt. %	SAMPLE NO 19	20	21	22	23
<b>Part A</b>					
Uvacure 1500	30	30	30	30	30
Microthene FP800-00	10	10	10	10	10
Airvol 203 S (PVOH)	10		10	10	10
Quickrete Concrete Mix		20			
DER 331					10
<b>Part B</b>					
Tone 0301 (Polyol)	29.65	29.65	29.65	29.65	29.65
Micropearls F30D		14.85			14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	8	5.5	5.5	5.5	5.5
Excpancel 091DU80	14.85				
Expancel 642WU			14.85	14.85	
Rxn Time		52 sec	55 sec	52 sec	54 sec
Rxn Temp	298 F	282 F	289 F	285 F	270 F
% Vertical Expansion	183%	282%	460%	450%	378%
Hardness – Shore A	75	18	10	12	43
Shrinkage	None	Very Slight	Very Slight	None	Noticeable

Components Wt. %	SAMPLE NO 24	25	26	27
<b>Part A</b>				
Uvacure 1500	30	20	30	30
Microthene FP800-00	10	10	10	

Airvol 203 S (PVOH)	10	10	10	10
Epalloy 8240		10		
Q325				10
<b>Part B</b> Tone 0301 (Polyol)		29.65	29.65	29.65
Micropearls F30D	14.85	14.85		14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5	5.5	5.5	5.5
Acclaim 6300	29.65			
Expancel 051DU			7.5	
Rxn Time	46 sec	55 sec	72 sec	50 sec
Rxn Temp		278 F	323 F	274 F
% Vertical Expansion	206%	388%	219%	570%
Hardness – Shore A	23	39	53	21
Shrinkage	None	Noticeable	Very Slight	Noticeable

Components Wt. %	SAMPLE NO 28	29	30	31	32
<b>Part A</b> Uvacure 1500		30	30	30	30
Microthene FP800-00	10	10	10	10	
Airvol 203 S (PVOH)	10	10	10	10	10
Sartomer K126	30				
Epon 58005			10		
DER 736				10	
Sodium Silicate					10
<b>Part B</b> Tone 0301 (Polyol)	29.65	29.65	29.65	29.65	29.65
Micropearls F30D	14.85		14.85	14.85	14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5	5.5	5.5	5.5	5.5
Expancel 820DU		14.85			
Rxn Time	50 sec	50 sec	54 sec	61 sec	58 sec
Rxn Temp	285 F	296 F	273 %	271 F	
% Vertical Expansion	483%	410%	396%	374%	410%
Hardness – Shore A	20	32	34	30	10
Shrinkage	None	Noticeable	Noticeable	Significant	Significant

Components Wt. %	SAMPLE NO 33	34	35	36
<b>Part A</b> Uvacure 1500	30	30	30	30
Microthene FP800-00		10	10	10
Airvol 203 S (PVOH)	10	10	10	10
Q Cel 650-D	10			
Texaphor Special		4		
Blue Dish Wish Soap			2	
<b>Part B</b> Tone 0301 (Polyol)	29.65	29.65	29.65	
Micropearls F30D	14.85	14.85	14.85	14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5	5.5	5.5	5.5
Expancel 820DU				
E-351 Polyol				29.65
Rxn Time	49 sec	68 sec	71 sec	40 sec
Rxn Temp	294 F		281 F	295 F
% Vertical Expansion	445%	530%	502%	302%
Hardness – Shore A	18	13	23	38
Shrinkage	None	Very Slight	None	None

Components Wt. %	SAMPLE NO 37	38	39	40	41
<b>Part A</b> Uvacure 1500	30	30	30	30	30
Microthene FP800-00	10	10	10	10	10
Airvol 203 S (PVOH)	10	10	10		10
Shell CMD 50809			5		
Z6124				2	
<b>Part B</b> Tone 0301 (Polyol)			29.65	29.65	29.65
Micropearls F30D	14.85	14.85	14.85	14.85	14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5	5.5	5.5	5.5	5.5
Sartomer SB 400	29.65				

LIR 403		14.65			
Santolink X1-100		15			
75% Isopropyl Alcohol					9.65
Rxn Time	37 sec	32 sec	55 sec		
Rxn Temp	292 F		305 F		
% Vertical Expansion	345%	188%	462%	561%	638%
Hardness – Shore A	39	45	32	15	3
Shrinkage	Noticeable	None	Very Slight	None	Very Slight

<b>Components Wt. %</b>	<b>SAMPLE NO</b>	<b>43</b>	<b>44</b>	<b>45</b>
<b>Part A</b>	<b>42</b>			
Uvacure 1500		30	30	25
Microthene FP800-00	10	10	10	10
Airvol 203 S (PVOH)	10	10	10	10
Uvacure 1502	30			
Expancel 461DU			14.85	
Epon 1510				5
<b>Part B</b>				
Tone 0301 (Polyol)	29.65	14.65	29.65	29.65
Micropearls F30D	14.85	14.85		14.85
H <sub>3</sub> PO <sub>4</sub> (Reagent:85%)	5.5			
Santolink X1-100		15		
Amberphos-54		6.5	6.5	6.5
Rxn Time	67 sec	45 sec	51 sec	82 sec
Rxn Temp	282 F		291 F	
% Vertical Expansion	450%	440%	340%	334%
Hardness – Shore A	25	43	35	43
Shrinkage	None	Very Slight	None	Noticeable

<b>Components Wt. %</b>	<b>SAMPLE NO</b>	<b>47</b>	<b>48</b>	<b>49</b>
<b>Part A</b>	<b>46</b>			
Uvacure 1500	30	30	30	30
Microthene FP800-00	10	10		
Airvol 203 S (PVOH)	10	10		
Erisys GE-60		10		



<b>SAT 200 (silyl)</b>			<b>20</b>	
<b>Kraton D1107</b>				<b>5</b>
<b>Part B</b> Tone 0301 (Polyol)	20			
Micropearls F30D	14.85	14.85	14.85	14.85
Amberphos-54	6.5			
#1 Castor Oil	9.65			
<b>Arcol LG-650</b>		<b>29.65</b>		
<b>HQ54 (73% H<sub>3</sub>PO<sub>4</sub>)</b>		<b>6.5</b>	<b>6.5</b>	
<b>Arcol DP-1022</b>			<b>29.65</b>	<b>29.65</b>
<b>H3PO4 (75% technical grade)</b>				<b>6.5</b>
Rxn Time	47 sec	<b>110 sec</b>	<b>101 sec</b>	<b>83 sec</b>
Rxn Temp	287 F		<b>289 F</b>	
% Vertical Expansion	364%	<b>350%</b>	<b>458%</b>	<b>620%</b>
Hardness – Shore A	32	<b>25</b>	<b>9</b>	<b>7</b>
Shrinkage	Very Slight	<b>Very slight</b>	<b>Noticeable</b>	<b>Significant</b>

### EXAMPLE 19

<b>Components wt.%</b>	<b>Sample No. 1</b>	<b>Sample No. 2</b>	<b>Sample No. 3</b>	<b>Sample No. 4</b>
<b>Part A</b>				
Uvacure 1500	30	30	30	30
Microthene FP 800-00	10	10	10	10
Airvol 203 S (PVOH)	10	10	10	10
<b>Part B</b>				
Tone 0301 (Polyol)				
Micropearls F30D	14.85	14.85	14.85	14.85
Amberphos-54 (AMMGA)	6.5	6.5	6.5	6.5
Tone 0201	14.65	20	9.65	
Santolink XI-100	15	9.65	20	
Arcol DP-1022				29.65
Initial Rxn Time	27 sec	24 sec	--	63 sec
Initial Rxn Temp	204 F	--	--	201 F
Rxn Time	42 sec	35 sec	--	99 sec
Rxn Temp	--	--	--	--
% Vertical Expansion	293 %	277 %	249 %	550 %
Hardness - Shore A	55	59	53	4
Shrinkage	Significant (> 2mm<4mm from edge of tin cup) after	Significant (> 2mm<4mm from edge of tin cup) after	Noticeable shrinkage (>1mm<2mm from edge of tin cup) after	Noticeable shrinkage (>1mm<2mm

	cooling to RT	cooling to RT	cooling to RT	from edge of tin cup) after cooling to RT
--	---------------	---------------	---------------	---

### EXAMPLE 20

Component wt%	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4	Sample No. 5	Sample No. 6
<b>Part A</b>						
Uvacure 1500	30	30	30	30	40	34.46
Nipol 1312			15	15		
Lucant HC-2000	15					
Carbopol EZ-1		5				
Microthene FP800-00		10				
Microthene FN-514-00					20	12.75
Kraton D1107						3.79
<b>Part B</b>						
Arcol PPG-425	20		15	10	20	
Micropearls F30D	14.85		14.85	14.85	14.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	6.5		6.5	6.5	6.5	6.5
Jeff P. Control Part B		50				
Arcol DP-1022			5	10		
Tone 0301						14.65
Santolink XI-100						15
Initial Rxn Time	40 sec	34 sec	51 sec	50 sec	51 sec	29 sec 31 sec
Final Rxn Time	50 sec	52 sec	65 sec	65 sec	73 sec	70 sec 65 sec
% Ht Expansion	390 %	423 %	387 %	310 %	292 %	374 %
Water Absorption	81 % 75 %	31 % 33 %	73 % 98 %	46 % 58 %	19 % 28 %	18 % (matl chunk)
Shore A Hardness	27	28	32	33	62	38
Shrinkage	0	0	≈ 5.5 mm	≈ 7 mm	2 mm	2 mm

Component wt%	Sample No. 7	Sample No. 8	Sample No. 9	Sample No. 10	Sample No. 11	Sample No. 12
<b>Part A</b>						
Uvacure 1500	30.4	34.46	34.46	50.7	50.7	27.9
Kraton D1107	3.34	3.79	3.79	5.6	5.6	3.1
Microthene FN514-00	11.26	12.75	12.75	18.8	18.8	10.3
<b>Part B</b>						
Tone 0301	14.65					
Santolink XI-100	15					
Micropearls F30D	14.85	14.85	11.6	14.85	14.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	6.5	6.5	5.1	6.5	12.5	6.5
Arcol PPG-425						20
Arcol DP-1022		29.65	23.3	29.65	29.65	
Initial Rxn Time		83 sec	96 sec	101 sec	28 sec	41 sec
Final Rxn Time		107 sec	132 sec	136 sec	42 sec	58 sec
% Ht Expansion	432 %	398 %	368 %	252 %	313 %	402 %



Density (g/in <sup>3</sup> )						
Water Absorption						
Shore A Hardness	36	13	18	28	24	46
Shrinkage	1 mm	1 mm	3 mm	5 mm	1 mm	0.5 mm

Component wt%	Sample No. 19	Sample No. 20	Sample No. 21	Sample No. 22	Sample No. 23	Sample No. 24
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	35
Microthene FN-514-00						
Kraton D1107						
Nipol 1312	15	15	15	15	15	15
Carbopol EZ-1		5	5			
Epon CMD 50859						5
<b>Part B</b>						
Arcol PPG-425	20	20	20	20	20	20
Micropearls F30D	24.85	24.85	24.85	24.85	24.85	24.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	8.5	8.5	8.5	8.5	8.5	8.5
Glycolic Acid (70% tech)	6		3			
#1 Castor Oil				8		
Z6040					1	
Initial Rxn Time	30 sec	30 sec	25 sec	33 sec	36 sec	37 sec
Final Rxn Time	42 sec	41 sec	39 sec	59 sec	57 sec	52 sec
% Ht Expansion	382 %	402 %	417 %	421 %	455 %	432 %
Density (g/in <sup>3</sup> )						
Water Absorption		144 % 133 %	152 % 162 %	66 % 55 %	105 % 108 %	99 % 129 % (open chunk: 137%)



Shore A Hardness	18	24	17	23	29	23
Shrinkage	3 mm	0 mm	1.5 mm	0 mm	0.5 mm	0 mm

Component wt%	Sample No. 25	Sample No. 26	Sample No. 27	Sample No. 28	Sample No. 29	Sample No. 30
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	40
Microthene FN-514-00						
Kraton D1107						
Nipol 1312	15					
SAT 030		15	15	15	15	
CN 301						15
<b>Part B</b>						
Arcol PPG-425		20	20	20	20	20
Micropearls F30D	24.85	24.85	24.85	24.85	24.85	24.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	8.5	8.5	8.5	8.5	8.5	8.5
Arcol Acclaim 6300	20					
#1 Castor Oil			8	15		8
Tone EC Monomer					10	
Initial Rxn Time	36 sec	38 sec	50 sec	61 sec	54 sec	42 sec
Final Rxn Time	--	56 sec	74 sec	95 sec	73 sec	60 sec
% Ht Expansion	150%	555 %	492 %	370 %	467 %	445 %
Density (g/in <sup>3</sup> )			1.939			
Water Absorption		67 % 95 % (open chunk: 260%)	48 % 45 % (open chunk: 49%)	44 % 37 %	50 % 95 % (open chunk: 75%)	62 % 39 % (open chunk: 143%)
Shore A Hardness		18	24	20	25	25
Shrinkage	1.5 mm	0 mm	0 mm	0.5 mm	3 mm	0 mm

Component wt%	Sample No. 31	Sample No. 32	Sample No. 33	Sample No. 34	Sample No. 35	Sample No. 36
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	
CN 301		15		15		
Uvacure 1534						40
<b>Part B</b>						
Arcol PPG-425	20		20	20	20	20
Micropearls F30D	24.85	24.85	24.85	24.85	24.85	24.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	8.5	8.5	8.5	8.5	8.5	8.5
#1 Castor Oil	8	8	8	8	8	8
CN 301	15		22		15	15
Ebecryl 81		20				
Z6040				1.5		
KR TTS					1.5	
Initial Rxn Time	41 sec	88 sec	38 sec	39 sec	37 sec	163 sec
Final Rxn Time	59 sec	120 sec	55 sec	--	54 sec	265+ sec
% Ht Expansion	461 %	153 %	386 %	479 %	433 %	
Density (g/in <sup>3</sup> )						
Water Absorption	99 % 66 %		37 % 45 %	45 % 34 %	294 % 243	



	(open chunk: 160%)		(open chunk: 151%)	(open chunk: 158%)	% (open chunk: 184%)	
Shore A Hardness	22	72	22	22	22	
Shrinkage	0 mm	1 mm	0 mm	0.5 mm	0 mm	

Component wt%	Sample No. 37	Sample No. 38	Sample No. 39	Sample No. 40	Sample No. 41	Sample No. 42
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	50.91
Ricon 100	15					19.09
SAT 030		25				
Ricon 184			15	15		
Expancel 461DU				10		
Actipol E-16					15	
<b>Part B</b>						
Arcol PPG-425	20	20	20	20	20	20
Micropearls F30D	24.85	24.85	24.85	14.85	24.85	24.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	8.5	8.5	8.5	8.5	8.5	8.5
#1 Castor Oil	8	8	8	8	8	8
Initial Rxn Time	39 sec	66 sec	39 sec	31 sec	39 sec	43 sec
Final Rxn Time	64 sec	102 sec	63 sec	50 sec	68 sec	72 sec
% Ht Expansion	504 %	348 %	470 %	345 %	395 %	432 %
Density (g/in <sup>3</sup> )						
Water Absorption	77 % 79 % (open chunk: 119%)	26 % 43 % (open chunk: 121%)	84 % 93 % (open chunk: 108%)	60 % 63 % (open chunk: 166%)	71 % 90 % (open chunk: 101%)	105 % 72 % (open chunk: 186%)
Shore A Hardness	26	22	27	30	32	31
Shrinkage	0 mm	0 mm	0 mm	0 mm	0 mm	0.5 mm

Component wt%	Sample No. 43	Sample No. 44	Sample No. 45	Sample No. 46	Sample No. 47	Sample No. 48
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	40
SAT 030	15		15	15	15	15
Kraton L-2203		15				
Erisys GE-35						10
<b>Part B</b>						
Arcol PPG-425	20	20	20			20
Micropearls F30D	24.85	24.85	24.85	24.85	24.85	24.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	8.5	8.5	8.5	8.5	8.5	8.5
#1 Castor Oil	8	8	8	8	8	8
CN 301	15					
Santolink XI-100			10			
Tone 0301				20	29.65	
Initial Rxn Time	52 sec	39 sec	53 sec	31 sec	36 sec	48 sec
Final Rxn Time	84 sec	65 sec	83 sec	49 sec	53 sec	77 sec
% Ht Expansion	275 %	431 %	350 %	480 %	502 %	395 %
Density (g/in <sup>3</sup> )						

Water Absorption	34 % 36 % (open chunk: 119%)	79 % 61 % (open chunk: 62 %)	26 % 40 % (open chunk: 117%)	159 % 220 % (open chunk: 212%)	102 % 102 % (open chunk: 205%)	96 % 75 % (open chunk: 135%)
Shore A Hardness	30	26	27	17	16	25
Shrinkage	0 mm	0 mm	0.5 mm	0 mm	0 mm	0 mm

Component wt%	Sample No. 49	Sample No. 50	Sample No. 51	Sample No. 52	Sample No. 53	Sample No. 54
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	40
SAT 030	15	15	15	15	15	15
Erisys GE-35	10					
<b>Part B</b>						
Arcol PPG-425	20	20		20	20	20
Micropearls F30D	24.85	24.85	24.85	24.85	24.85	24.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	12	8.5	8.5	8.5	8.5	8.5
#1 Castor Oil	8	8	8	8		8
Kraton L-2203		15				
Tone 0201			20			
Santicizer 261				10	15	
Z6124						2
Z6040						1
Initial Rxn Time	29 sec	59 sec	27 sec	51 sec	53 sec	45 sec
Final Rxn Time	50 sec	118 sec	36 sec	67 sec	63 sec	78 sec
% Ht Expansion	410 %	317 %	422 %	263 %	225 %	458 %
Density (g/in <sup>3</sup> )	1.826					
Water Absorption	74 % (open chunk: 53% & 64%)	29 % 44 % (open chunk: 86%)	104 % 138 % (open chunk: 133%)	19 % 22 % (open chunk: 75%)	41 % 54 % (open chunk: 137%)	22 % 24 % (open chunk: 180%)
Shore A Hardness	12	34	30	44	50	23
Shrinkage	0 mm	0.5 mm	2 mm	0 mm	0 mm	0.5 mm

Component wt%	Sample No. 55	Sample No. 56	Sample No. 57	Sample No. 58	Sample No. 59	Sample No. 60
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	40
SAT 030				15		
SAT 010	15					
SAT 200		15				
PBD 605			15		15	15
<b>Part B</b>						
Arcol PPG-425	20	20	20	20	20	15
Micropearls F30D	24.85	24.85	24.85	24.85	24.85	24.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	8.5	8.5	8.5	8.5	8.5	8.5
#1 Castor Oil	8	8	8	8	8	8
Santicizer 261				5	5	10
Z6124				2	2	2
Z6040				1	1	1



Initial Rxn Time	58 sec	50 sec	40 sec	51 sec	40 sec	29 sec
Final Rxn Time	83 sec	76 sec	80 sec	85 sec	69 sec	39 sec
% Ht Expansion	444 %	435 %	536 %	388 %	433 %	320 %
Density (g/in <sup>3</sup> )						
Water Absorption	44 % 60 % (open chunk: 191%)	96 % 105 % (open chunk: 131%)	25 % 27 % (open chunk: 34%)	65 % 53 % (open chunk: 85%)	32 % 33 % (open chunk: 54%)	115 % 105 % (open chunk: 197%)
Shore A Hardness	25	20	24	22	23	19
Shrinkage	0 mm	0 mm	0 mm	0 mm	0.5 mm	0.5 mm

Component wt%	Sample No. 60	Sample No. 61	Sample No. 62	Sample No. 63	Sample No. 64	Sample No. 65
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	40
PBD 605	15	20	25	15	15	15
<b>Part B</b>						
Arcol PPG-425	20	20	20	20	25	20
Micropearls F30D	24.85	24.85	24.85	24.85	24.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	8.5	8.5	8.5	8.5	8.5	6.5
#1 Castor Oil	6	6	6	3	8	
Santicizer 261	7	7	7	5		
Z6124	2	2	2			
Z6040	1	1	1			
Initial Rxn Time	37 sec	43 sec	43 sec	42 sec	46 sec	48 sec 42 sec
Final Rxn Time	62 sec	74 sec	72 sec	71 sec	86 sec	77 sec 60 sec
% Ht Expansion	411 %	357 %	337 %	493 %	423 %	475 % 486 %
Density (g/in <sup>3</sup> )						2.17
Water Absorption	28 % 26 % (open chunk: 119%)	17 % 25 % (open chunk: 126%)	56 % 34 % (open chunk: 121%)	43 % 49 % (open chunk: 134%)	29 % 26 % (open chunk: 71%)	17 % 15 % (open chunk: 14%)
Shore A Hardness	23	22	18	20	20	29 30
Shrinkage	0.5 mm	0.5 mm	0.5 mm	0.5 mm	0.5 mm	0.8 mm

Component wt%	Sample No. 66	Sample No. 67	Sample No. 68
<b>Part A</b>			
Uvacure 1500	40	40	40
PBD 605	15	15	15
Z6040			1
<b>Part B</b>			
Arcol PPG-425	20	20	20
Micropearls F30D	24.85	24.85	24.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	8.5	8.5	8.5
#1 Castor Oil	3	3	3
Santicizer 261	7	7	7
Z6124		2	2
Initial Rxn Time	39 sec	38 sec	41 sec
Final Rxn Time	65 sec	55 sec	56 sec
% Ht Expansion	455 %	403 %	439 %
Density (g/in <sup>3</sup> )			

Water Absorption	57 % 72 % (open chunk=153%)	131 % 130 % (open chunk=160%)	107 % 88 % (open chunk=156%)
Shore A Hardness	28	25	28
Shrinkage	0.5 mm	0.5 mm	1 mm

### EXAMPLE 21

Component wt%	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4	Sample No. 5	Sample No. 6
<b>Part A</b>						
Uvacure 1500	32.7	36.4	43.6	40	40	40
PBD 605	12.3	13.6	16.4	20	25	15
<b>Part B</b>						
Arcol PPG-425	20	20	20	20	20	20
Micropearls F30D	14.85	14.85	14.85	14.85	14.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	6.5	6.5	6.5	6.5	6.5	6.5
Santicizer 160						4
Initial Rxn Time	47 sec	47 sec	50 sec	51 sec	54 sec	49 sec
Final Rxn Time	75 sec	70 sec	78 sec	--	104	67 sec
% Ht Expansion	564 %	454 %	430 %	427 %	387 %	426 %
Density (g/in <sup>3</sup> )						
H <sub>2</sub> O Absorption 1						
H <sub>2</sub> O Absorption 2						
H <sub>2</sub> O Absorption 3	37 %	31 %	14 %	28 % 30 %	42 % 31 %	74 %
Shore A Hardness	28	31	32	33	33	38
Shrinkage	0 mm	0.3 mm	1.5 mm	0.8 mm	0.5 mm	3 mm

Component wt%	Sample No. 7	Sample No. 8	Sample No. 9	Sample No. 10	Sample No. 11	Sample No. 12
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	40
PBD 605	15	15	15	10	15	15
<b>Part B</b>						
Arcol PPG-425	20	20	20	20	20	20
Micropearls F30D	14.85	14.85	14.85	14.85	14.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	6.5	6.5	6.5	6.5	8	9.5
Santicizer 160	8					
Santicizer 278		4	8			
Initial Rxn Time	51 sec	54 sec	55 sec	42 sec	32 sec	27 sec
Final Rxn Time	71 sec	80 sec	83 sec	57 sec	--	38 sec
% Ht Expansion	332 %	395 %	338 %	529 %	567 %	500 %
Density (g/in <sup>3</sup> )						
H <sub>2</sub> O Absorption 1		44 % 23 %	26 % 26 %	61 % 90 %	25 % 33 %	56 % 65 %
H <sub>2</sub> O Absorption 2						
H <sub>2</sub> O Absorption 3	106 %	63 %	87 %	126 %	34 %	92 % 83 %
Shore A Hardness	43	24	32	30	29	28
Shrinkage	4 mm	2 mm	2 mm	0 mm (had radial shrinkage)	0.5 mm (had radial shrinkage)	0.75 mm (had radial shrinkage)



Component wt%	Sample No. 13	Sample No. 14	Sample No. 15	Sample No. 16	Sample No. 17	Sample No. 18
<b>Part A</b>						
Uvacure 1500	40	40	40	40	40	40
PBD 605	15	15	15	15	15	15
Epon SU2.5	10					
Epon 828				5		
<b>Part B</b>						
Arcol PPG-425	20		29.98		20	20
Micropearls F30D	14.85	14.85	22.27	14.85	14.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	6.5	6.5	9.75	6.5	6.5	
CAPA 316		20				
Tone 0301				20		
Dicy 1400				2	0.26	
85% H <sub>3</sub> PO <sub>4</sub> (tech)						6.5
Initial Rxn Time	--	20 sec	50 sec	74 sec	49 sec	36 sec
Final Rxn Time	--	--	--	99 sec	70 sec	76 sec
% Ht Expansion	414 %	430 %	445 %		451 %	433 %
Density (g/in <sup>3</sup> )						
H <sub>2</sub> O Absorption 1			97 % 106 %			19% 14 %
H <sub>2</sub> O Absorption 2						
H <sub>2</sub> O Absorption 3	40 % 56 %	69% 65% 57%	128 %			22% 13% 17%
Shore A Hardness	53	43	26		30	32
Shrinkage	3 mm	0 mm (had radial shrinkage)	2.8 mm	very significant	2.6 mm	0 mm

Component wt%	Sample No. 19	Sample No. 20	Sample No. 21	Sample No. 22	Sample No. 23	Sample No. 24
<b>Part A</b>						
Uvacure 1500	40	40	35	35	35	40
PBD 605	15	15	15	15	15	15
Epon 828						10
<b>Part B</b>						
Arcol PPG-425	20					20
Micropearls F30D	14.85	14.85	14.85	14.85	14.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	6.5	6.5	6.5	5	8	6.5
Ebecryl 170	5					15
Desmophen L-951		20	20	20	20	
Initial Rxn Time	25 sec	44 sec 50 sec	48 sec 48 sec	72 sec	37 sec 34 sec	18 sec
Final Rxn Time	43 sec	64 sec 67 sec	79 sec 70 sec	100 sec	57 sec 51 sec	35 sec
% Ht Expansion	415 %	467 % 510 %	505 % 442 %	420 %	530 % 535 %	289 %
Density (g/in <sup>3</sup> )	1.93	2.02				
H <sub>2</sub> O Absorption 1	9 % 15 %	15% 18%	20 % 22 %		39 % 19 %	
H <sub>2</sub> O Absorption 2					36 %	
H <sub>2</sub> O Absorption 3	39 % 22 %	32% 22% 37%	60% 49%		66% 55%	81 % 48 %



			73%		61%	
Shore A Hardness	35	38 31	29 32	31	30 27	52
Shrinkage	0.3 mm	1.5 mm 0.8 mm	0.5mm 0.75mm	3.3 mm	0.5mm 0.75mm	2 mm

Component wt%	Sample No. 25	Sample No. 26	Sample No. 27	Sample No. 28	Sample No. 29	Sample No. 30
<b>Part A</b>						
Uvacure 1500	40	30	40	30	40	40
PBD 605	15		15	15	15	7.5
Epon 828				10		
SAT 030						7.5
<b>Part B</b>						
Arcol PPG-425						20
Micropearls F30D	14.85	14.85	14.85	14.85	14.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)			6.5	6.5	6.5	6.5
Desmophen L-951	20		10	20		
Ebecryl 170	20	30	5	5		
Santicizer 261			10			
K-Flex 188					20	
Initial Rxn Time		< 10 sec	21 sec	25 sec	27 sec	48 sec
Final Rxn Time			36 sec	41 sec	--	61 sec
% Ht Expansion		350 %	358 %	333%	456 %	
Density (g/in <sup>3</sup> )				3.45		
H <sub>2</sub> O Absorption 1				28 % 38 %		
H <sub>2</sub> O Absorption 2				89 %		
H <sub>2</sub> O Absorption 3			94 % 95 %	84% 83% 85%	32% 28% 30%	19 % 22 %
Shore A Hardness			39	37	47	
Shrinkage		0.5 mm	2.5 mm	2.5 mm	0.9 mm	

Component wt%	Sample No. 31	Sample No. 32	Sample No. 33	Sample No. 34	Sample No. 35	Sample No. 36
Part A						
Uvacure 1500	40	40	40	40	40	20
PBD 605	7.5		15	15		15
Epon 828						
SAT 030	7.5					
Trilene M-101		15				
Hycar 1300x40					15	
Uvacure 1533						20
Part B						
Arcol PPG-425	20	20	20	20	20	20
Micropearls F30D	14.85	14.85	14.85	14.85	14.85	14.85
75% H <sub>3</sub> PO <sub>4</sub> (tech)	6.5	6.5	6.5	6.5	6.5	6.5
Ebecryl 170	2					
#1 Castor Oil			6			
Santicizer 141				10		
Initial Rxn Time	31 sec	46 sec	49 sec	56 sec	45 sec	47 sec

Final Rxn Time	49 sec	60 sec	68 sec	71 sec	55 sec	78 sec
% Ht Expansion		394 %				
Density (g/in <sup>3</sup> )						
H <sub>2</sub> O Absorption 1						
H <sub>2</sub> O Absorption 2						
H <sub>2</sub> O Absorption 3	22 % 23 %	114% 81% 107%	17 % 15 %	51 % 70 %		30 % 34 %
Shore A Hardness		40				
Shrinkage		2.75 mm				

Component wt%	Sample No. 37	Sample No. 38	Sample No. 39	Sample No. 40	Sample No. 41	Sample No. 42
<b>Part A</b>						
Uvacure 1500	25	40	40	40		
PBD 605	15	15	15	15		
Epon 828					45	45
Uvacure 1534	15					
<b>Part B</b>						
Arcol PPG-425	20	20	20	20		
Micropearls F30D	14.85	14.85	14.85	14.85	15	15
75% H <sub>3</sub> PO <sub>4</sub> (tech)	6.5					
H <sub>3</sub> PO <sub>4</sub> (conc: >95%)		6.5	4.9	4	10	10
Tone EC Monomer					20	
#1 Castor Oil						20
Initial Rxn Time	58 sec	28 sec	33 sec	37 sec	58 sec	33 sec
Final Rxn Time	73 sec	37 sec	52 sec	50 sec	93 sec	--
% Ht Expansion		274 %	261 %	181 %	510 %	300 %
Density (g/in <sup>3</sup> )					1.8	
H <sub>2</sub> O Absorption 1						
H <sub>2</sub> O Absorption 2						
H <sub>2</sub> O Absorption 3	27 % 33 %		15 % 20 % (72 hrs)		37 % 38 % (72 hrs)	
Shore A Hardness		62	60	62	48	68
Shrinkage					0 mm	0 mm

Component wt%	Sample No. 43	Sample No. 44	Sample No. 45	Sample No. 46
<b>Part A</b>				
Epon 828	45	45	50	50
Vertrel XF				5
<b>Part B</b>				
Micropearls F30D	15	15	15	10
H <sub>3</sub> PO <sub>4</sub> (conc: >95%)	10	20	10	10
Santicizer 261	20			
Pure Grain Alcohol (EtOH)		20		
Santicizer 97			20	20
Initial Rxn Time	24 sec	108 sec	27 sec	26 sec
Final Rxn Time	72 sec	130 sec	--	--
% Ht Expansion	182 %	575 %	376 %	297 %



Density (g/in <sup>3</sup> )		1.37		
H <sub>2</sub> O Absorption 1				
H <sub>2</sub> O Absorption 2				
H <sub>2</sub> O Absorption 3		843 % (after 15 mins)		
Shore A Hardness	78	4		62
Shrinkage	0.5 mm	0.5 mm		0 mm

### EXAMPLE 22

Component wt%	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4	Sample No. 5	Sample No. 6
<b>Part A</b>						
Uvacure 1500	40					
PBD 605	15					
Epon 828		40	40	35	35	50
Santicizer 97				5	5	
<b>Part B</b>						
Desmophen L-951	20					
H <sub>3</sub> PO <sub>4</sub> (75% tech)	6.5					
Micropearls F30D	2	10	10	10	10	10
H <sub>3</sub> PO <sub>4</sub> (>95% conc)		10	10	10	10	10
Arcol PPG-425		20				
Santicizer 97			20	20	15	20
#1 Castor Oil					5	
Initial Rxn Time	43 sec	263 sec	20 sec	19 sec	24 sec	25 sec
Final Rxn Time	54 sec	299 sec	--	--	--	--
% Ht Expansion	86 %	308 %	189 %	118 %	122 %	184 %
Density (g/in <sup>3</sup> )		2.50	6.20		6.93	6.13
H <sub>2</sub> O Absorption 1	3 %					
H <sub>2</sub> O Absorption 2						
H <sub>2</sub> O Absorption 3	3 %	27 % 23 %	8 % 10 %	18% 22% 18%	11 % 12 %	5 % 6 %
Shore A Hardness			88	90	88	88
Shore D Hardness (##) = calculated	(32)		27 (30)	30 (32)	27 (30)	28 (30)
Shrinkage	2.2 mm	0 mm	0 mm	0 mm	0 mm	0 mm

Component wt%	Sample No. 7	Sample No. 8	Sample No. 9	Sample No. 10	Sample No. 11	Sample No. 12
<b>Part A</b>						
Epon 828	40	20	40	40	40	60
CMD 50859		20				
Tone EC					2	3
<b>Part B</b>						
Santicizer 97		20	20	15	15	15
H <sub>3</sub> PO <sub>4</sub> (>95% conc)	10	10	10	10	10	10
Micropearls F30D	10	10	6	6	4	4



Santicizer 160	20					
#1 Castor Oil				5	5	5
Initial Rxn Time	17 sec	22 sec	18 sec	22 sec	22 sec	30 sec
Final Rxn Time	--	--	--	--	--	--
% Ht Expansion	260 %	152 %	120 %	145 %	99 %	92 %
Density (g/in <sup>3</sup> )		4.20	7.03	6.40	6.94	8.19
H <sub>2</sub> O Absorption 1						
H <sub>2</sub> O Absorption 2						
H <sub>2</sub> O Absorption 3	40% 32% 39%	50 % 34 %	13 % 12 %	13 % 11 %	15 % 10 %	3 % 2 %
Shore A Hardness		80	91	86	93	98
Shore D Hardness (##) = calculated		23 (22)	32 (33)	26 (28)	34 (36)	46 (42)
Shrinkage	0 mm	0 mm	0 mm	0 mm	0 mm	0 mm

Component wt%	Sample No. 13	Sample No. 14	Sample No. 15	Sample No. 16	Sample No. 17	Sample No. 18
<b>Part A</b>						
Epon 828	40	60	80	80	50	50
Santicizer 97	5	7.5	10	10		
Vertrel XF				5	10	
<b>Part B</b>						
Santicizer 97	15	15	15	15	20	20
H <sub>3</sub> PO <sub>4</sub> (>95% conc)	10	10	10	10	10	10
Micropearls F30D	6	6	6	6	5	10
#1 Castor Oil	10	10	10	10		10
Initial Rxn Time	--	--	69 sec	66 sec	24 sec	41 sec
Final Rxn Time	--	--	--	--	--	--
% Ht Expansion	--	--	60 %	61 %	238 %	123 %
Density (g/in <sup>3</sup> )	--	--	--	--	--	4.64-8.38
H <sub>2</sub> O Absorption 1	--	--	--	--	--	--
H <sub>2</sub> O Absorption 2	--	--	--	--	--	--
H <sub>2</sub> O Absorption 3	14 %	8 % 8 %	1 % 1 %	--	--	32 % 35 %
Shore A Hardness	--	--	--	90	--	90
Shore D Hardness (##) = calculated	--	--	--	(32)	--	29 (32)
Shrinkage	--	--	0 mm	0 mm	0 mm	0 mm

Component wt%	Sample No. 19
<b>Part A</b>	
Epon 828	65
Santicizer 97	5
<b>Part B</b>	
Santicizer 97	15
H <sub>3</sub> PO <sub>4</sub> (>95% conc)	10
Micropearls F30D	10
Initial Rxn Time	25 sec
Final Rxn Time	--

% Ht Expansion	176 %
Density (g/in <sup>3</sup> )	6.55
H <sub>2</sub> O Absorption 1	
H <sub>2</sub> O Absorption 2	
H <sub>2</sub> O Absorption 3	4 % 4 % (after 48 hrs)
Shore A Hardness	93
Shore D Hardness (## = calculated)	(36)
Shrinkage	0 mm

### EXAMPLE 23

This Example illustrates the ability to tailor the inventive foam compositions and obtain foams having a wide range of characteristics. A foam of relatively low density was produced by in accordance with Example 18. The foam was obtained by combining the following foam precursors:

#### Part A:

AMOUNT	COMPONENT	TRADE NAME	SUPPLIER
18.2g	cylcoaliphatic epoxy	Uvacure 1500	Radcure
1.8g	phenoxy resin	Phenoxy PKHP-200	Paphen
30g	bis-A epoxy	D.E.R. 736	Dow Chemical

#### Part B

AMOUNT	COMPONENT	TRADE NAME	SUPPLIER
29.65g.	polyester polyol	Tone 0301	Union Carbide
14.85 g.	vinylidene chloride	Micropearls F30D	Pierce & Stevens
	encapsulated n-butane		
5.5g.	phosphoric Acid (85%)		ACROS

Each component (Part A & B) was individually mixed by hand using a hand driven paddle in a cup or ointment can. The two were brought together in a single vessel, again mixed by hand, and allowed to react. The foam produced was similar in appearance to other types listed above, but had a final specific gravity 0.16 g/ml.

## EXAMPLE 24

The following Example demonstrates employing the inventive foam as a structural material between two laminates to fabricate furniture. The components listed in the following Table were combined in accordance with Examples 18-22.

5

Components wt. %	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4
<b>Part A</b>				
Epon 828	75	--	--	50
Santicizer 97	5	--	--	--
Epon 813	--	50	--	--
Epon 825	--	--	50	--
<b>Part B</b>				
Santicizer 97	20	20	20	23
H <sub>3</sub> PO <sub>4</sub> (>95% conc)	10	10	10	10
Micropearls F30D	10	10	10	10
Initial Rxn Time	40 sec	33 sec	28 sec	28 sec
Final Rxn Time	--	--	--	--
% Ht Expansion	136%	262%	225%	170%
Density 1 (g/in <sup>3</sup> )	--	--	--	--
Density 2 (g/in <sup>3</sup> )	8.77	--	5.70	5.52
H <sub>2</sub> O Absorption 1	1% 1% 2%	--	--	--
H <sub>2</sub> O Absorption 2	--	--	--	--
Shore A Hardness	95	--	--	81
Shore D Hardness	40	--	--	
(##) = calculated	(38)			(23)
Shrinkage	0 mm	0 mm Had significant radial shrinkage	0 mm	0 mm

The following Table lists components employed in the above Table for making foam.

Trade Name	Component	Supplier
#1 Castor Oil	#1 Castor Oil	Commercial
Epon 825	Bis A Epoxy	Shell Chemical Co.
Epon 828	Bis A Epoxy	Shell Chemical Co.
Epon 813	Bis A Epoxy Modified: (74% Bisphenol A epichlorohydrin resin & 26% Cresyl glycidyl ether)	Shell Chemical Co.
Santicizer 97	Dialkyl Adipate	Solutia
H <sub>3</sub> PO <sub>4</sub> (>95% conc)	Phosphoric acid: Took Harcros 75% Technical Grade & distilled to >90% acid concentration	DeNOVUS
Micropearls F30D	Thermal Blowing Agent: isobutane encapsulated in polymer vinylidene chloride	HM Royal (Pierce & Stevens)

10

Sample No. 1 above was combined and introduced into a mold comprising standard 1"x4"x12" boards and laminating materials comprising wood-grain Formica® and fiber-

reinforced paper board that were maintained a defined distance about by wood spacers, i.e., a distance of about ½ inch. The boards and laminating materials were placed into “C” clamps and a vise. The foam composition was prepared and poured between the laminating materials. Once the foam reaction was completed and the foam had cooled to room temperature, the assembly was visually inspected. The foam had adhered to the laminating materials and provided structural support.

A skilled person in this art would understand that these exemplary processes can be modified by manipulating process variables such as time and temperature of each aforementioned mixing step, mixing rate (RPM), time under vacuum, radiation source (e.g., UV light) and length of exposure and distance from source, and level of vacuum (mm Hg) as well as operating a continuous process. While the above Examples illustrate a batch process a skilled person in this art after having reviewed and understood the instant disclosure, would be capable of manipulating the aforementioned process variables to tailor the instant composition for a virtually unlimited array of product applications.

While the present invention has been described in certain preferred embodiments thereof, it will be apparent that various substitutions, omissions, modifications, and other changes which may be made without departing from the spirit of the invention. Thus, the present invention should be limited only by the scope of the following claims including equivalents thereof.

## CLAIMS

1. A method for producing a foam comprising:
  - (a) combining at least one epoxy component with at least one acid source  
5 component and at least one encapsulated blowing agent under conditions sufficient to provide an exothermic reaction; and
  - (b) utilizing heat from the exothermic reaction so as to expand the combined components to form a foam.
- 10 2. The method according to Claim 1 further comprising (c) recovery of the foam.
3. The method according to Claim 1 wherein said at least one acid source is substantially water free.
- 15 4. The method according to Claim 1 wherein the epoxy component is present in a first precursor composition and the acid source is present in a second precursor composition.
- 20 5. The method according to Claim 4 wherein the first precursor composition further comprises a blowing agent comprising at least one member selected from the group consisting of butane, propane, isopentane and fluorocarbons.
6. The method according to Claim 4 wherein the second precursor composition further comprises a carrier material.
- 25 7. The method according to Claim 1 wherein the epoxy compound and the acid source are present in a single foam precursor composition.
8. The method according to Claim 7 wherein the acid source comprises at least one photoinitiator.

9. The method according to Claim 1 wherein step (a) occurs at least in part in a containment device.

10. The method according to Claim 9 wherein the containment device comprises  
5 polyethylene, polyester, vinyl, ethylene vinyl acetate, nylon, ethylene vinyl acetate, styrene-  
isoprene-styrene, styrene-butadiene-styrene or other blocked copolymers, polybutadiene,  
polyamide, modified EVA's, modified polyethylene, modified polybutadiene, GMA, SBR or  
mixtures thereof.

10 11. The method according to Claim 1 wherein the acid source comprises phosphoric acid.

12. A foam composite comprising a foam according to Claim 1 which is at least partially  
in contact with at least one member selected from the group consisting of polyethylene,  
polyester, vinyl, ethylene vinyl acetate, nylon, ethylene vinyl acetate, styrene-isoprene-styrene  
15 block copolymers, styrene-butadiene-styrene block copolymers, polybutadiene, polyamide,  
modified EVA's, modified polyethylene, modified polybutadiene, GMA, SBR or mixtures  
thereof.

13. The foam composite of Claim 12 further comprising at least one of polyethylene or  
20 styrene powders.

14. A foam precursor comprising:

(a) an A-side foam precursor composition comprising an epoxy compound, and an  
encapsulated blowing agent, and;

25 (b) a B-side foam precursor composition comprising an acid source.

15. The foam precursor according to Claim 14 wherein (a) further includes a modifying  
material.



16. The foam precursor according to Claim 14 wherein (b) further comprises a carrier material.

17. The foam precursor of Claim 14 wherein said acid source is substantially free of water.

18. The foam precursor of Claim 14 wherein the encapsulated blowing agent comprises a thermoplastic shell that contains a butane blowing agent.

19. The foam precursor of Claim 14 wherein at least one of the A-side precursor and the B-side precursor further comprises castor oil, at least one benzyl phthalate and at least one member selected from the group consisting of Bis A epoxy and Bis F epoxy.

20. The foam precursor of Claim 14 wherein said epoxy compound is a bis-A or bis-F epoxy compound; the blowing agent is a butane blowing agent and the A-side precursor further comprises at least one member selected from the group consisting of polypropylene, polyethylene and polyvinyl alcohol.

ABSTRACT OF THE DISCLOSURE

5 A low-temperature foam compositions and that are produced from an epoxy compound and an acid source can be substantially free of polyurethane or isocyanate chemistry. The disclosed compositions and precursors thereof reduce, if not eliminate, the presence of conventional undesirable compounds and by-products thereof.

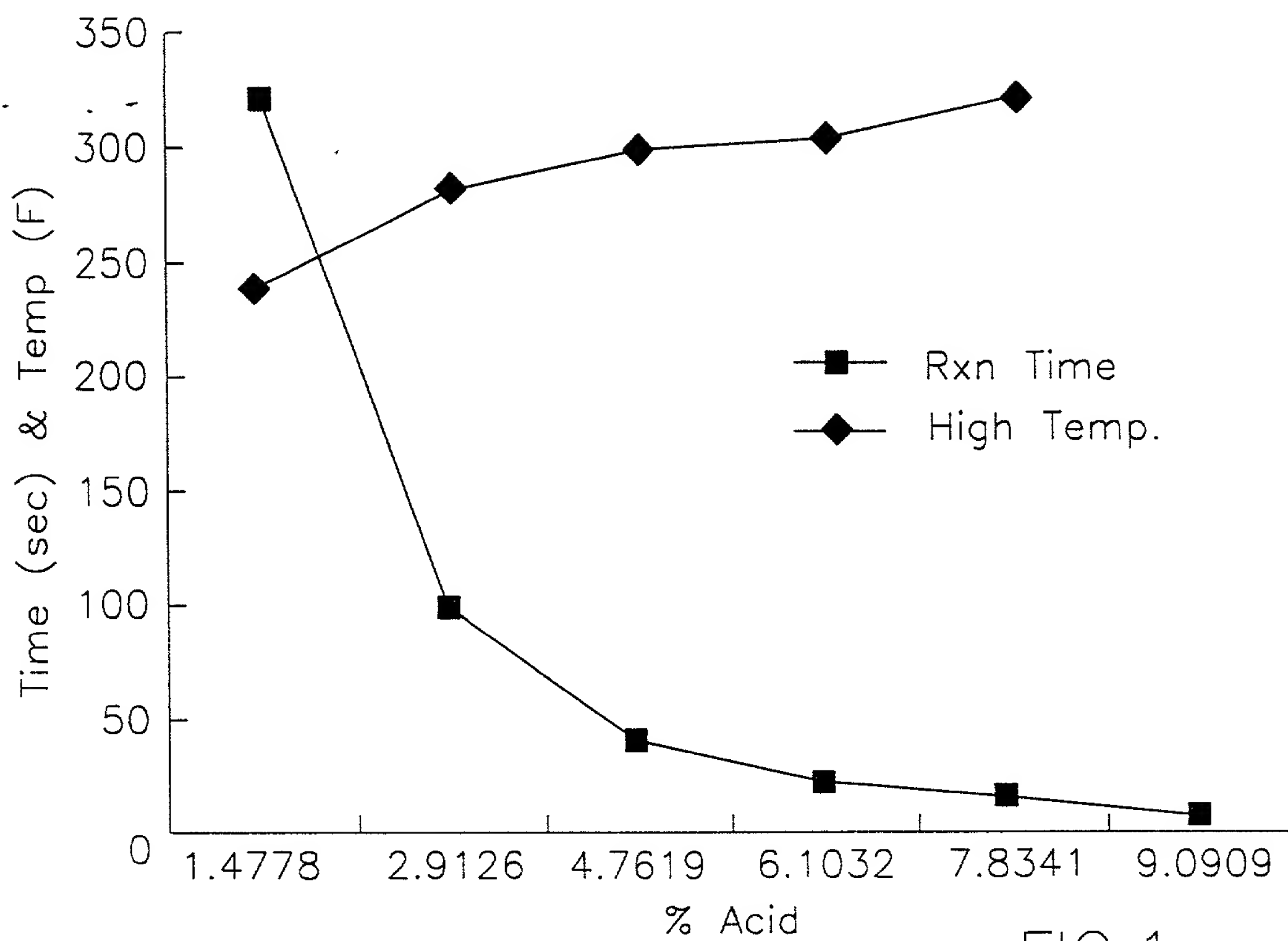


FIG.1

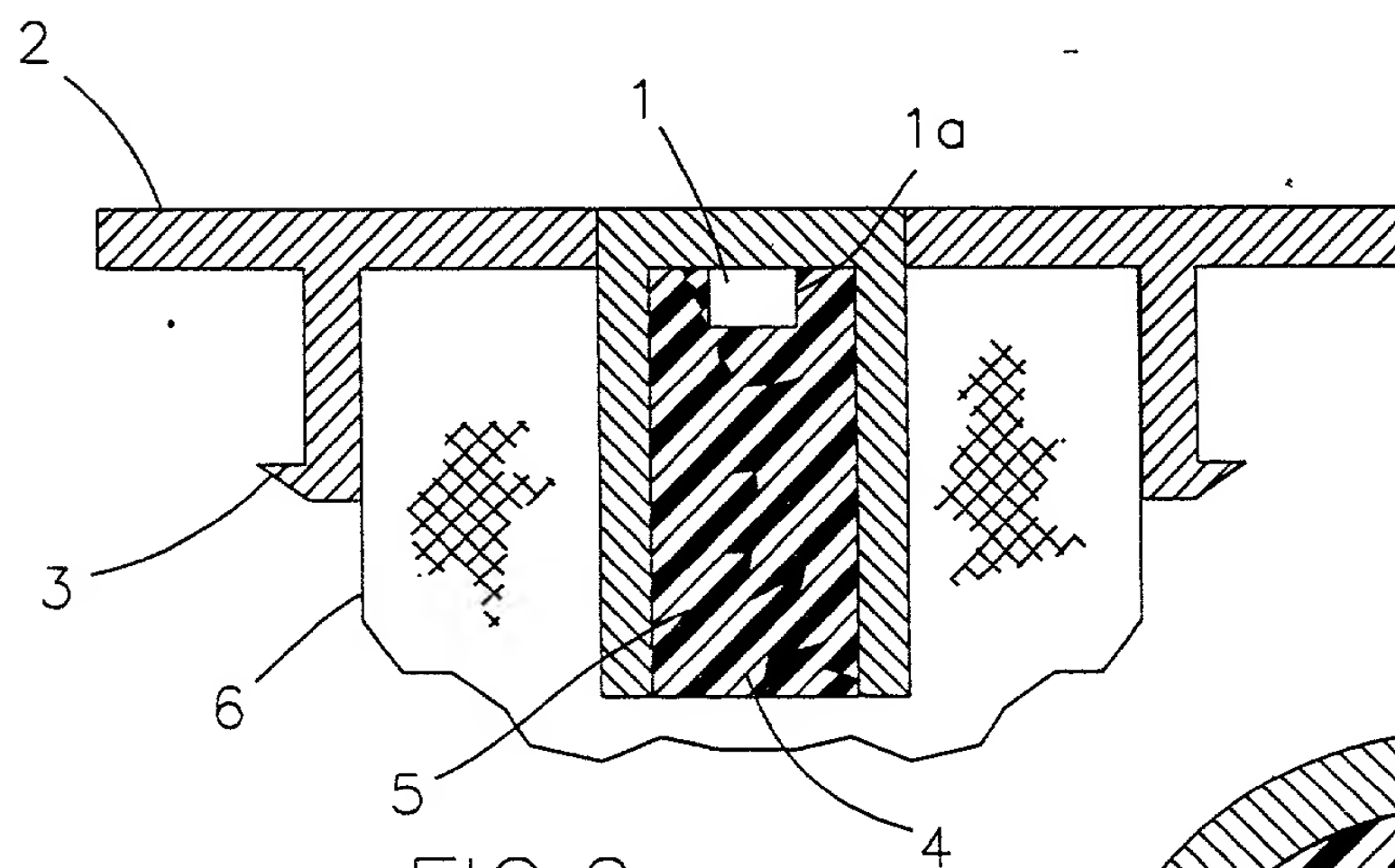


FIG.2a

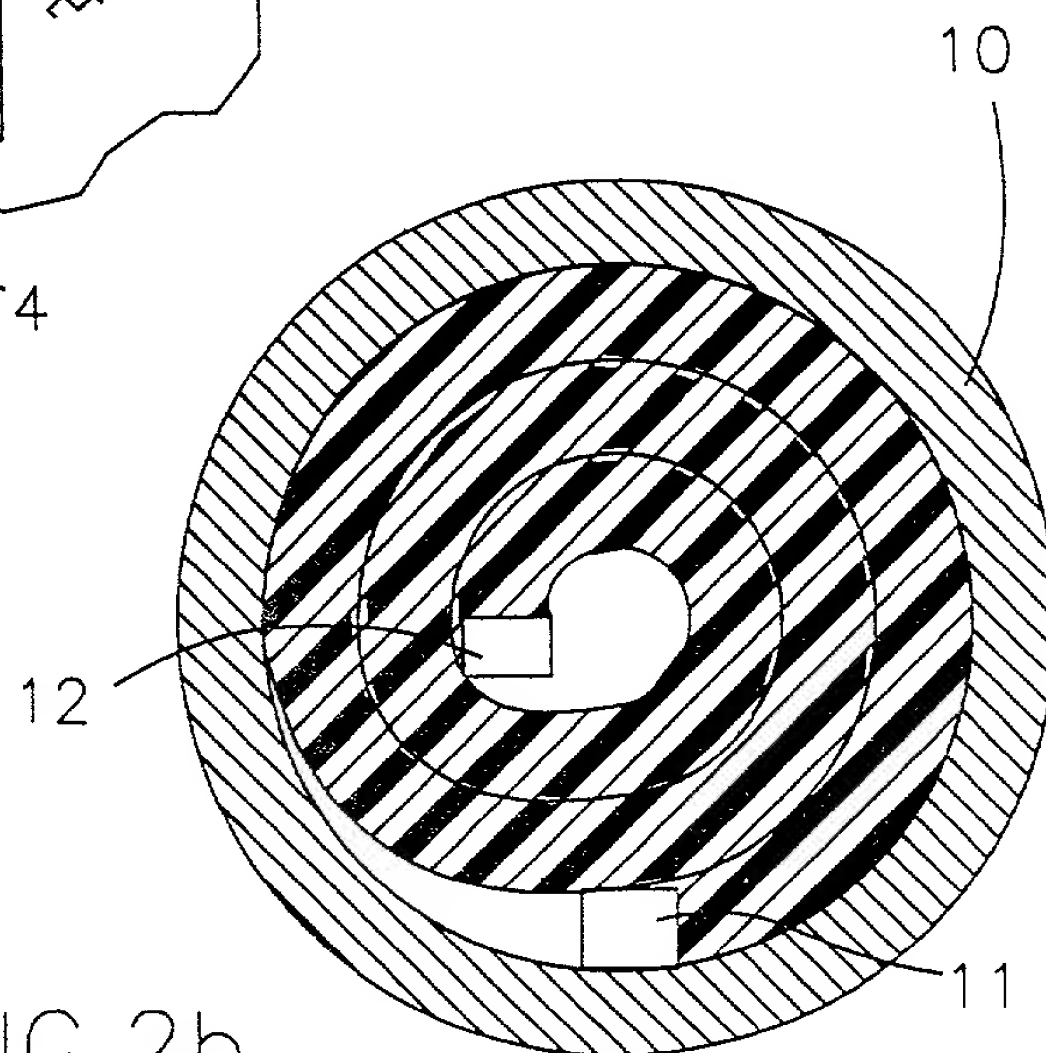


FIG.2b

Docket No.  
OB003JP-3

## Declaration and Power of Attorney For Patent Application

### English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

" NOVEL FOAMING COMPOSITIONS AND METHODS FOR MAKING AND USING THE COMPOSITIONS "

the specification of which

(check one)

☒ is attached hereto.

☐ was filed on \_\_\_\_\_ as United States Application No. or PCT International

Application Number \_\_\_\_\_

and was amended on \_\_\_\_\_

(if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) or Section 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Not Claimed

(Number)

(Country)

(Day/Month/Year Filed)

☐

(Number)

(Country)

(Day/Month/Year Filed)

☐

(Number)

(Country)

(Day/Month/Year Filed)

☐

I hereby claim the benefit under 35 U.S.C. Section 119(e) of any United States provisional application(s) listed below:

<u>60/047,273</u>	<u>MAY 21, 1997</u>
(Application Serial No.)	(Filing Date)
<u>60/079,205</u>	<u>MARCH 24, 1998</u>
(Application Serial No.)	(Filing Date)
<u>                    </u>	<u>                    </u>
(Application Serial No.)	(Filing Date)

I hereby claim the benefit under 35 U. S. C. Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112, I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C. F. R., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

<u>09/081,967</u>	<u>MAY 20, 1998</u>	<u>PENDING</u>
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
<u>09/197,124</u>	<u>NOVEMBER 20, 1998</u>	<u>PENDING</u>
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
<u>                    </u>	<u>                    </u>	<u>                    </u>
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

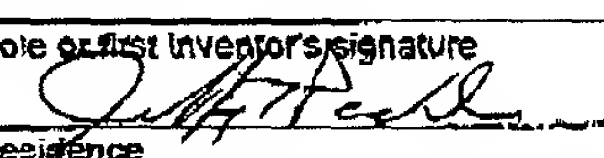
**POWER OF ATTORNEY:** As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. *(list name and registration number)*

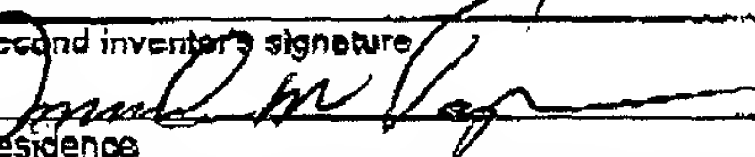
**MICHAEL K. BOYER** (USPTO REG NO 33,085)

**DOUGLAS E. SITTLER** (USPTO REG NO 37,722)

Send Correspondence to: **MICHAEL K. BOYER**  
**ORSCHELN MANAGEMENT CO**  
**2000 US HWY 63 SOUTH**  
**MOBERLY, MISSOURI, 65270, U.S.A.**

Direct Telephone Calls to: *(name and telephone number)*  
**MICHAEL BOYER 660 269-4536 FAX X4530 email: mboyer@orscheln.com**

Full name of sole or first inventor	
<b>JEFFREY T. PACHL</b>	
Sole or first inventor's signature	Date
	4/28/99
Residence	
<b>15214 N.E. 188TH STREET, HOLT, MISSOURI, 64048</b>	
Citizenship	
<b>USA</b>	
Post Office Address	
<b>SAME AS RESIDENCE</b>	

Full name of second inventor, if any	
<b>DONALD W. TAYLOR</b>	
Second inventor's signature	Date
	4/28/99
Residence	
<b>1129 CAMELOT HILLS, LIBERTY, MISSOURI, 64068</b>	
Citizenship	
<b>USA</b>	
Post Office Address	
<b>SAME AS RESIDENCE</b>	



Full name of third inventor, if any <b>JAMES W. FREITAG</b>	Date <b>4/28/99</b>
Third inventor's signature <i>James W. Freitag</i>	
Residence <b>206 CARLA STREET, EXCELSIOR SPRINGS, MISSOURI, 64024</b>	<b>12624 French Ridge Kearney, Mo. 64060</b>
Citizenship <b>USA</b>	
Post Office Address <b>SAME AS RESIDENCE</b>	

Full name of fourth inventor, if any	
Fourth inventor's signature	Date
Residence	
Citizenship	
Post Office Address	

Full name of fifth inventor, if any	
Fifth inventor's signature	Date
Residence	
Citizenship	
Post Office Address	

Full name of sixth inventor, if any	
Sixth inventor's signature	Date
Residence	
Citizenship	
Post Office Address	